

Bulletin 157

DEPARTMENT OF THE INTERIOR

FRANKLIN K. LANE, SECRETARY

BUREAU OF MINES

VAN. H. MANNING, DIRECTOR

INNOVATIONS IN THE METALLURGY
OF LEAD

BY

DORSEY A. LYON

AND

OLIVER C. RALSTON



ENGINEERING

ENGINE STORAGE

WASHINGTON
GOVERNMENT PRINTING OFFICE
1918

DEPARTMENT OF THE INTERIOR

FRANKLIN K. LANE, SECRETARY

BUREAU OF MINES

VAN. H. MANNING, DIRECTOR

INNOVATIONS IN THE METALLURGY
OF LEAD

BY

DORSEY A. LYON

AND

OLIVER C. RALSTON



WASHINGTON
GOVERNMENT PRINTING OFFICE
1918

The Bureau of Mines, in carrying out one of the provisions of its organic act—to disseminate information concerning investigations made—prints a limited free edition of each of its publications.

When this edition is exhausted copies may be obtained at cost price only through the Superintendent of Documents, Government Printing Office, Washington, D. C.

The Superintendent of Documents *is not an official of the Bureau of Mines*. His is an entirely separate office and he should be addressed:

SUPERINTENDENT OF DOCUMENTS,
Government Printing Office,
Washington, D. C.

The general law under which publications are distributed prohibits the giving of more than one copy of a publication to one person. The price of this publication is 20 cents.

First edition. July, 1918.

CONTENTS

	Page.
Introduction.....	9
Scope of research work.....	9
Problems in treating low-grade and complex lead ores.....	11
Oxidized ores of lead.....	12
Materials tested.....	14
Methods of treatment.....	16
Screen analyses of materials containing lead carbonate.....	17
Leaching of lead from carbonate ores.....	19
Preliminary tests.....	20
Use of acidified brine for leaching.....	23
First series of preliminary tests.....	23
Second series of preliminary tests.....	25
Third series of preliminary tests.....	27
Tests of tailing from Wilbert dump.....	27
Tests of material from Scranton mine.....	30
Effect of sulphates in brines.....	31
Preliminary tests.....	31
Other tests.....	32
Cyclic leaching.....	34
Laboratory leaching tests.....	34
Procedure and results.....	34
Discussion of results of tests.....	36
Conclusions from laboratory tests.....	38
Large-scale cyclic leaching.....	38
Plant used and method of operating.....	39
Results of tests and discussion of results.....	41
Purifying the precipitate.....	43
Precipitation with lime.....	43
Precipitation on scrap iron.....	44
Precipitation with sodium sulphide and hydrogen sulphide.....	44
Precipitation with sodium carbonate.....	44
Effect of using limestone.....	44
Tests to determine best conditions for maximum extraction.....	45
Effect of volume of brine used.....	45
Effect of heating the pulp.....	46
Effect of adding chloride of lime.....	47
Construction of plant.....	47
Materials used in plant construction.....	48
Character of sample tested.....	49
Conclusions from results of tests.....	49
Cyclic leaching and electrolytic precipitation.....	50
Roasting and leaching test at Salt Lake station.....	50
Use of iron anode.....	51
Tests at Bunker Hill and Sullivan plant.....	52
Leaching unroasted material with brine containing ferric chloride.....	52
Conclusions on leaching and electrolytic precipitation of lead.....	57
Chemical reactions involved in brine leaching.....	57

Sulphide ores of lead—Continued.	
Complex sulphides containing lead—Continued.	
Lead-zinc ores and concentrates—Continued.	Page.
Conclusions as to experiments with lead-zinc-sulphide concentrates.	157
Recovery of copper and silver.	158
Limitations of the method.	158
Lead-iron middlings and complex sulphides.	159
Character of middlings.	160
Tests of middlings from a Missouri mill.	161
Results of tests.	161
Tests of material from Bullion Coalition mine in Utah.	163
Test of material from Bingham mine in Utah.	165
Experiments with material from North Star dump, Hailey, Idaho.	166
Materials from mines near Pine Creek, Idaho.	167
Comparison with electrolytic method.	167
General summary.	168
Publications on mineral technology.	170
Publications available for free distribution.	170
Publications that may be obtained only through the Superintendent of Documents.	170
Index.	173

TABLES.

TABLE 1. Results of analyses of material containing oxidized lead.	13
2. Mineralogical association of lead in materials tested.	15
3. Screen analyses of oxidized lead ores.	18
4. Results of screen analysis of material from Horn Silver dump.	19
5. Results of screen analysis of material from Wilbert dump.	19
6. Solubility of lead chloride.	20
7. Results of leaching oxidized lead ore from May Day mine with dilute solution of hydrochloric acid.	21
8. Results of leaching lead carbonate ores in acidified brine.	24
9. Results of preliminary tests of leaching lead carbonate ores with acidified brines.	25
10. Results of leaching Wilbert tailing with acidified brine.	29
11. Results of leaching Scranton ore with acidified brine.	31
12. Results showing effect of sodium sulphate on solubility of lead sulphate in brine solutions.	31
13. Results of cyclic leaching of material from Horn Silver tailing dump with acidified brine and precipitation with lime.	35
14. Results of preliminary tests to determine proportion of acid needed.	41
15. General results of large-scale tests of cyclic leaching.	41
16. Results of assays of precipitate, tailing, and solution from cyclic leaches.	42
17. Results of tests showing the effect of the ratio of solution to ore on the extraction obtained.	46
18. Results of tests showing effect of heating pulp on the extraction of lead.	46
19. Results of tests made to improve leach tailings.	47
20. Results of cyclic leaching and electrolysis of Horn Silver tailings.	51
21. Data on leaching tests made with and without iron in the solution.	53
22. Results of acid brine leaches of natural oxidized ores.	55

TABLE 23. Results of shaft roasting of argentiferous lead carbonate ore from the Colorado mine.....	Page. 62
24. Results of shaft roasting of argentiferous lead carbonate ore from the American Flag mine.....	63
25. Results of shaft roasting of material from Wilbert tailings dump....	65
26. Results of volatilization of lead chloride from Bullionville tailing...	70
27. Results of volatilization and leaching tests of Dry Valley tailing...	70
28. Data on tests of material from Nevada United mine, Fly, Nev.....	76
29. Data on tests of material from Dry Valley mill dump, Pioche, Nev.	77
30. Data on tests of material from Horn Silver dump, Frisco, Utah....	77
31. Data on tests of material from Bingham Mines Co., Bingham, Utah.	78
32. Data on tests of material from Yellow Pine mine, Goodsprings, Nev.	78
33. Data on tests of material from Trapper mill dump, Melrose, Mont...	78
34. Data on tests of material from Hayden mill feed, Clarkdale Ariz....	79
35. Data on tests of material from Sells mine, Alta, Utah.....	79
36. Results of sulphidizing material from May Day mine with dry hydrogen sulphide.....	89
37. Results of sulphidizing material from Chief Consolidated mine with dry hydrogen sulphide.....	90
38. Results of sulphidizing Daly-Judge carbonate ore with dry hydrogen sulphide.....	91
39. Results of sulphidizing ore from Shattuck mine with sodium sulphide.....	97
40. Results of sulphidizing ore from Scranton mine and material from Wilbert mill dump.....	98
41. Results of sulphidizing material from Bullion Becks lime dump with sodium sulphide.....	99
42. Average results of sulphidizing miscellaneous materials.....	100
43. Results of sulphidizing ore from May Day mine with calcium polysulphide.....	104
44. Results of cyclic leaching of calcines.....	129
45. Results of brine leaching of calcine containing 10.7 per cent lead...	131
46. Results of analyses of zinc concentrates.....	141
47. Data on roasting for tests of mixed flotation concentrate.....	144
48. Volatilization data for tests of mixed flotation concentrate.....	145
49. Data on lead extraction in tests of mixed flotation concentrates....	146
50. Data on zinc extraction in tests of mixed flotation concentrate....	147
51. Data on copper extraction in tests of mixed flotation concentrate...	148
52. Summary of data on extraction of metals in tests of mixed flotation concentrate.....	148
53. Data on roasting for tests of electrostatic feed and concentrate.....	149
54. Volatilization data for tests of electrostatic feed and concentrate....	150
55. Data on lead extraction in leaching tests of electrostatic feed and concentrate.....	151
56. Data on zinc extraction in leaching tests of electrostatic feed and concentrate.....	152
57. Data on copper extraction in leaching tests of electrostatic feed and concentrate.....	153
58. Summary of data on extraction of metals in tests of electrostatic feed and concentrate.....	154
59. Analyses of lead-iron middlings from mills in southern Missouri....	160
60. Analyses of mixed-sulphide materials.....	161
61. Results of tests of middlings from Bonne Terre mill in southeastern Missouri.....	162

ILLUSTRATIONS.

	Page.
FIGURE 1. Curve showing solubility of lead chloride (PbCl_2).....	20
2. Curves showing solubility of lead chloride in brine.....	22
3. Curves showing relation of lead extraction to consumption of acid in first series of leaching tests with acidified brine.....	26
4. Curves showing relation of lead extraction to consumption of acid in second series of leaching tests with acidified brine.....	27
5. Curve showing effects of time on lead extraction and acid efficiency in leaching Wilbert tailing with acidified brine.....	28
6. Flow-sheet of test plant.....	39
7. Sketch of laboratory suction filter.....	40
8. Proposed flow sheet for lead-leaching plant.....	48
9. Sections of Holt-Dern roaster.....	59
10. Details of laboratory roaster of Holt-Dern type.....	61
11. Christensen down-draft laboratory roaster.....	68
12. Flow sheet of proposed mill for leaching oxidized ores of lead.....	107
13. Flow sheet of mill for volatilization of lead.....	116

INNOVATIONS IN THE METALLURGY OF LEAD.

By DORSEY A. LYON AND OLIVER C. RALSTON.

INTRODUCTION.

The data reported in this bulletin are largely the result of experiments conducted by the Salt Lake City station of the Bureau of Mines in cooperation with the department of metallurgical research of the University of Utah. The work on lead is only one of the phases of the investigations carried on at that station during the years 1914 to 1916. The results of similar work with zinc ores and with copper and other ores will be published in future reports of the bureau.

SCOPE OF RESEARCH WORK.

The scope of the work done at the station is shown by the following extract from a report published by the University of Utah.^a

Although Utah has long been a metal producer her high standing as a mining State is due at the present time to the large bodies of low-grade ore found within her boundaries, a notable example of which are the mammoth deposits of monzonite-porphphy at Bingham, which are being mined by the Utah Copper Co. However, the successful treatment of these low-grade ores has presented a problem which has not as yet been satisfactorily solved in a great many instances. It is due to this fact that at its tenth regular session in 1913, the legislature of the State provided for the establishment of a metallurgical research department in connection with the State school of mines of the University of Utah. As stated in the act (Laws of Utah, 1913, ch. 102, sec. 2, pp. 199-200), providing for this department, the purposes of this research department have been "to conduct experiments and research, either alone or in cooperation with the National Bureau of Mines and other agencies, with a view of finding ways and methods of profitably treating low-grade ores, of obtaining other information that shall have for its object the benefit of the mining industry and the utilization and conservation of the mineral resources of the State, and to publish and distribute bulletins and articles relating to the department and its work.

This act became effective in July, 1913. For the remainder of that year the research work was temporarily directed by Prof. Robert H. Bradford, head of the department of metallurgy of the University of Utah. In January, 1914, a working agreement was entered into with the United States Bureau of Mines. By the terms of this agreement the work of the metallurgical research department is under the direction of metallurgists of the Bureau of Mines assigned to duty at the university and Salt Lake City. From January, 1914, to July, 1916, Mr. D. A. Lyon, metallurgist, was

^a Wells, A. E., Report of the department of metallurgical research; Bull. 9, Utah Eng. Exp. Station. Univ. of Utah, 1917, pp. 4-8.

in charge of the work, assisted by other members of the metallurgical staff of the bureau assigned to duty at Salt Lake City. After organizing the work of the station at the University of Utah and supervising the research problems for nearly three years, Mr. Lyon was assigned to duty at the new station in the Northwest.

The staff at present assigned by the Bureau of Mines to the department consists of: The metallurgist in charge, a metallurgist, an assistant metallurgical engineer, a metallurgical assistant, a geologist and microscopist, a junior chemist, a chief clerk, a junior clerk, and a stenographer.

The University of Utah is providing the building and the greater part of the equipment, and also five metallurgical research fellowships of the yearly value of \$720 each. The fellowships are awarded to graduates of colleges, preferably of mining schools, who have shown special aptitude for research investigations. Their employment extends over the entire twelve months.

The administrative head of the department is the director of the State school of mines. No employee of the United States Bureau of Mines has anything to do with the handling of the funds appropriated by the State for supporting the department.

The laboratory work of the department is for the most part carried on in the metallurgical building. Due to this fact the department is in close touch with those in charge of the work in mining and metallurgy that is being carried on by the university for the benefit of undergraduate students doing work in these subjects. Moreover, it has received valuable assistance from those in charge of this work, and in this connection grateful acknowledgment is made of the help given by Dr. Robert H. Bradford, professor of metallurgy, and Robert S. Lewis, professor of mining, to those in charge of the work of the department of metallurgical research. Without their help, and the untiring efforts of the director of the State school of mines, the department would not have been able to accomplish the work it has done.

The fellows selected by the university authorities for the fiscal year 1913-14, and the problems assigned to them, were: L. F. Pattison, A. B., University of Utah (Hydrometallurgy of copper); W. G. Woolf, A. B., University of Utah (Hydrometallurgy of zinc); O. H. Pierce, A. B., University of Nebraska (Metallurgy of copper); A. E. Gartside, A. B., University of Oklahoma (Hydrometallurgy of zinc); and C. Y. Pfoutz, E. M., University of California (Hydrometallurgy of silver).

Messrs. Pattison, Gartside, and Pfoutz resigned their fellowships at the end of the fiscal year, and those selected, from a large number of applicants, to take their places for the fiscal year 1914-15, and the problems assigned to them, were: R. M. Isham, Ph. D., Columbia University (Smoke problems); C. L. Larson, E. M., University of Minnesota (Hydrometallurgy of lead); and H. J. Morgan, A. M., Stanford University (Flotation of oxidized ores).

Messrs. Isham and Pierce resigned shortly after entering the department and their places were taken by Messrs. Herbert J. Cutler, of the University of Michigan and Frank Cameron, of the University of Utah.

The fellows selected for the year 1915-16, and the problems assigned to them, were: Marvin J. Udy, University of Utah (Hydrometallurgy of lead); Richard W. Johnson, University of North Dakota (Hydrometallurgy of zinc); Clarence E. Sims, University of Illinois (Electrolytic precipitation of metals); Glenn L. Allen, University of Kansas (Flotation of oxidized lead ores); Harper C. Neeld, University of Nevada (Concentration of oxidized zinc ores); and George F. Stott, University of Utah (Milling losses in Utah).

The fellows appointed for the fiscal year 1916-17, and the problems on which they are working, are as follows: Edward J. Atckison, University of Oregon (Ore flotation problems); Theodore Erickson, University of Utah (Utah hydrocarbons); Charles W. Frith, University of Utah (concentration of oxidized zinc ores); Arthur J. McChrystal, resident of Eureka, Utah, Stanford University (treatment of complex sulphides); Grover J. Holt, University of North Dakota (hydrometallurgy of lead);

Orel E. Young, Case School of Applied Science (hydrometallurgy of zinc); Leonard D. Yundt, University of Utah (flotation oils); Clyde E. Williams, University of Utah (igneous concentration of lead).

Beside the work initiated and conducted directly by the department of metallurgical research, cooperative work has been carried on with institutions, and mining and metallurgical companies, who have had problems similar to those upon which the department was working. In these cases, men have been sent to work in the laboratories of the department by the cooperating concerns, these men carrying on their investigations under the same rules as are applied to the 'fellows' of the department. The discoveries which they make are the property of the department to be given to the people of the State through publications and otherwise.

As cooperators the department has the following: Utah State Conservation Commission. (This institution subscribes to the salary of an analyst in the departmental laboratory for examination of material brought in by prospectors of the State); McKeever Brothers, of New York; Prince Consolidated Mining Co., of Pioche, Nevada; Big Indian Copper Co., of Utah; Grand Central Mining Co., of Utah; Zinc Concentrating Co., of Boston; Utah Minerals Concentrating Co., of Eureka, Utah; United States Forest Products Laboratory, Madison, Wisconsin; and The General Engineering Co., of Salt Lake City.

Also, the following individuals have cooperated: E. H. Synder, of Pioche, Nevada; E. T. Cannon, of Salt Lake City; Dr. W. L. Ellerbeck, of Nephi, Utah; Prof. H. K. Benson, University of Washington; and C. T. Van Winkle, of Salt Lake City.

At the present time the annual appropriation made by the United States Bureau of Mines for the work in connection with this station amounts to about \$15,000, and the authorized appropriation of the State of Utah is \$7,500. Also, outside cooperating agencies are spending considerable money for the specific work carried on by them in this department. At the time of writing this report, five men, employed by cooperating concerns, are at work in the laboratories.

The Bureau of Mines has established at the University of Utah probably the best equipped microscopic laboratory in the West for metallurgical research work. This microscopic and metallographic examination work is being conducted by Dr. F. B. Laney, who was detailed to the bureau by the United States Geological Survey.

Below are shown the amounts that have been expended by the United States Bureau of Mines and the State of Utah in conducting the cooperative work which is being carried on by the department of metallurgical research.

Period.	U. S. Bureau of Mines.		University of Utah.	
	Salaries.	Supplies, etc.	Salaries.	Supplies, etc.
July 1, 1913-June 30, 1914.....	\$3,070.00		\$3,346.00	\$407.72
July 1, 1914-June 30, 1915.....	6,510.00	\$310.00	3,575.58	2,624.47
July 1, 1915-June 30, 1916.....	7,870.00	2,967.19	7,355.56	5,170.56
July 1, 1916-June 30, 1917.....	18,511.33	3,729.00	3,681.47	4,339.05

PROBLEMS IN TREATING LOW-GRADE AND COMPLEX LEAD ORES.

Three main problems in the treatment of lead ores had troubled metallurgists for many years and were still largely unsolved at the time the Salt Lake City station was established. These problems were as follows:

1. The treatment of lead carbonate ores with or without gold and silver.
2. The treatment of complex sulphides of lead and zinc either with or without metals other than lead or zinc.
3. The recovery of sulphide of lead from the finely divided "slimes" of the concentrating mills.

The third problem, that of prevention of losses in slimes, was being solved in a number of mills at the time the station was established. Flotation methods were just beginning to attract the interest of the general public. After some investigation it was decided that the most useful work of this sort that the station could perform would be the gathering and dissemination of information relating to the flotation process, rather than testing samples of the slimes submitted, because the flotation industry seemed to be in a healthy condition and the mill operators were willing to do their own testing in the attempts to prevent losses. Nearly every lead concentrating mill of any size that formerly suffered serious losses of galena in slimes has now a flotation unit to increase the recovery of metal.

OXIDIZED ORES OF LEAD.

In nearly every mining district where lead is found to any extent, the lead in the upper or weathered parts of the ore deposits, generally above the level of the ground water, is oxidized, being usually in the form of the carbonate, and occasionally the sulphate of lead. In most of the Western States the lead carbonate is, as a rule, accompanied by silver and occasionally by some gold or copper. Much of this "carbonate" ore has been very rich, owing to natural concentration by weathering. Such ores because of their richness and their needing for the most part no roasting before smelting have contributed greatly to the development of the mines.

Wherever the "carbonate" ore has been low-grade, however, preliminary concentration has been necessary. In milling such ore, serious losses in the tailings often have taken place, owing to the well-known tendency of lead carbonate to "slime" by breaking into thin flakes that float away with the gangue. The concentrates obtained by gravity concentration have usually been of good grade and in demand by the smelters. Dumps of these slime tailings abound in almost every important base-metal mining district of the western United States, and in regions where oxidation extends to any depth there are vast quantities of such low-grade ores from which the high-grade ore has been gouged out. In fact, in many districts the custom has been to mine only the ore of smelting grade and to leave the lower grade material in the mine. That ample supplies of low-grade oxidized ores of lead are available and that much of this material is being wasted can not be doubted.

TABLE 1.—Results of analyses of material containing oxidized lead.

[Analyst, E. F. Barrett.]

Mine from which sample was taken.	District.	State.	Material.	Insoluble.	Fe.	Al ₂ O ₃ .	CaO.	MgO.	CO ₂ .	S.	Zn.	Pb.	Cu.	Ag.	Au.	Mn.
				Per cent.	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.	Oz. per ton.	Oz. per ton.	Per cent.
Wilberl.....	Arco.	Idaho.	Tailing dump.	86.67	0.84	3.10	1.64		2.20	0.36		5.31		0.75		
Scranton.....	North Tintic.	Utah.	Mine ore.	16.25	16.25	7.60	2.67		1.81	0.40	0.87	9.61	0.10	1.32	Tr.	
May Day.....	Eureka.	do.	do.	81.90	1.30	Tr.	1.39		.52	.71	.50	4.30	Tr.	2.18	0.04	
Do.....	do.	do.	Tailing dump.	8.00	4.80	3.80	.40					3.21	.10	2.58	0.02	
Colorado.....	do.	do.	Mine ore.	80.80	1.19	8.70	.57		1.05		.50	3.50	Tr.	7.90	.08	
Do.....	do.	do.	do.	51.52	12.47	8.80	4.90		7.40		1.00	2.60	Tr.	9.55	.04	
Iron Blossom.....	do.	do.	do.	59.60	15.53	4.30	.50		.67		2.65	2.60	.20	6.88	.07	
Chief Consolidated.....	do.	do.	do.	61.30	4.91	1.05	6.59			.95	4.56	2.90	.80	4.70		
Eureka Hill.....	do.	do.	Slime dump.	82.80	4.91	3.47	4.15	3.52	7.40	.30	2.40	2.00	.40	7.20	.02	
Buffton Beck.....	do.	do.	do.	82.80	3.58	2.39	2.83		.50	.50	2.08	3.30	.40	6.80	.02	
Godiva.....	do.	do.	do.	66.20	5.00	5.21	4.81					1.72		5.90	.02	
Michigan-Tahiti.....	Alta.	do.	Mine ore.	46.68	5.48	6.92	12.94					3.46		5.90	.02	
Do.....	do.	do.	Shipping ore.	25.52	5.43	6.10	16.75					3.82		5.90	.02	
Do.....	do.	do.	Mine ore.	11.00	1.07	3.87	26.00					1.43		12.00	Tr.	
Do.....	do.	do.	do.	32.44	3.40	9.44	14.35					1.30		7.48	.06	
Do.....	do.	do.	do.	7.10	.79	Tr.	18.80	25.10			1.77	1.72		2.12	.06	
Do.....	do.	do.	do.	12.18	.94	Tr.	13.20	25.65			1.77	1.72		2.12	.06	
Do.....	do.	do.	do.	12.52	.94	.88	19.40	25.90			2.94	3.44		2.06	.06	
Do.....	do.	do.	do.	6.62	1.98	8.90	15.75	25.90			6.04	3.44		2.06	.06	
Do.....	do.	do.	Tailing dump.	53.60	3.40	8.50	2.20		1.37	0.15	6.04	7.80		2.06	.06	
Ontario.....	Park City.	do.	do.	79.46	3.30	5.28	.66		1.56	2.01	3.32	3.92		2.06	.06	
American Flag.....	do.	do.	Mine ore.	73.64	3.30	4.40	4.28		3.24	.40	3.32	3.92		2.06	.06	
Daily West.....	do.	do.	Tailing dump.	73.64	3.30	6.08	7.24		14.10	.60	3.32	3.92		2.06	.06	
Daily Judge.....	do.	do.	do.	43.30	3.30	3.20	3.20		2.33	.15	3.32	3.92		2.06	.06	
Nevada United.....	do.	do.	Mine ore.	11.60	37.60	21.20	1.00	.51		1.15	3.32	3.92		2.06	.06	
May King.....	Ely.	Nevada.	do.	11.60	37.60	21.20	1.00	.51		1.15	3.32	3.92		2.06	.06	
Princess Consolidated.....	Grand-primes.	do.	do.	11.60	37.60	21.20	1.00	.51		1.15	3.32	3.92		2.06	.06	
Do.....	Pueblo.	do.	do.	11.60	37.60	21.20	1.00	.51		1.15	3.32	3.92		2.06	.06	
Bullion Hill.....	do.	do.	Tailing dump.	11.60	37.60	21.20	1.00	.51		1.15	3.32	3.92		2.06	.06	
Do.....	do.	do.	do.	11.60	37.60	21.20	1.00	.51		1.15	3.32	3.92		2.06	.06	
Chances Delmonico.....	do.	do.	do.	11.60	37.60	21.20	1.00	.51		1.15	3.32	3.92		2.06	.06	
La Motte.....	do.	do.	do.	11.60	37.60	21.20	1.00	.51		1.15	3.32	3.92		2.06	.06	
Copper Queen Mining Co.....	do.	do.	Mine ore.	13.32	11.88	21.45	.66			3.01	1.00	14.00		18.20	.30	
Shattuck.....	do.	do.	do.	75.70	2.90	5.83	1.18			1.25	.86	14.00		18.20	.30	
Do.....	do.	do.	do.	66.65	11.22	.66	.66			.62	.26	1.18		1.18	.07	
Rayden Development Co.....	Charlebois.	do.	Tailing.	71.65	10.48		.93	.84				1.18		1.18	.07	

MATERIALS TESTED.

The results of analyses of representative samples of ores, tailings, and slimes containing oxidized lead are shown in Table 1. All of these materials are not completely oxidized, as some of them contain noteworthy amounts of lead sulphide. The sample from the tailings dump of the Horn Silver mine contained a larger proportion of the lead as the sulphate, anglesite; also the rather high zinc content consisted of the sulphide, sphalerite. Hence a further classification of these materials may be made, as has been done on page 168, when the individual peculiarities of each are considered with regard to its adaptability to various methods of treatment.

Mineralogical analysis of such oxidized materials is difficult, as they are often so "ocherous" that individual minerals can not be distinguished, whereas the minerals usually have few characteristics by which they can be distinguished optically from each other. It was found that an idea of the mineralogical association of the lead could be obtained by treating the material with certain solutions. A saturated solution of brine or a neutral solution of ammonium acetate, if used in excess, will dissolve all of the lead sulphate in the material; hence, this method was used for determining that constituent. After such treatment acidified saturated brine (salt solution) or acid ammonium acetate solution will quickly dissolve all of the lead carbonate. If the material being treated is left too long in contact with the solution, some of the sulphide of lead will also be dissolved, and a short time of contact with the warmed solution was found advisable. As acidified brine was used in one of the proposed methods of treatment, this figure was of practical value. Few minerals of lead other than the sulphate and the carbonate occur in such material in commercial amounts; hence, no other tests were necessary. The molybdate of lead, wulfenite, and the chlorophosphate, pyromorphite, can occasionally be found in small amounts. In material from the Nevada United mine the dioxide of lead, plattnerite, was also identified.

The results of mineralogical analyses of some of the materials tested with respect to the lead by the method described in the preceding paragraph are shown in Table 2.

TABLE 2. *Mineralogical association of lead in materials tested.*

[ANALYST, M. L. LEE.]

Name.	Material.	Pb. found cent. (1, 2, 3, 4, 5, 6, 7, 8, 9, 10, 11, 12, 13, 14, 15, 16, 17, 18, 19, 20, 21, 22, 23, 24, 25, 26, 27, 28, 29, 30, 31, 32, 33, 34, 35, 36, 37, 38, 39, 40, 41, 42, 43, 44, 45, 46, 47, 48, 49, 50, 51, 52, 53, 54, 55, 56, 57, 58, 59, 60, 61, 62, 63, 64, 65, 66, 67, 68, 69, 70, 71, 72, 73, 74, 75, 76, 77, 78, 79, 80, 81, 82, 83, 84, 85, 86, 87, 88, 89, 90, 91, 92, 93, 94, 95, 96, 97, 98, 99, 100).	Percentage of total Pb. present as PbS (insoluble in acid ammonium acetate solution).	Percentage of total Pb. insoluble in acidified solutions.
Chief Consolidated.....	Mine ore.....	5.12	56	4
Horn Silver.....	Tailings.....	7.4	14	0
Iron Blossom, sample 3....	Mine ore.....	3.5	22	9
Iron Blossom, sample X....	do.....	2.1	65	0
American Flag.....	do.....	4.06	17	0
Daly Judge.....	Carbonate ore.....	16.54	92	4
May Day.....	Tailings.....	3.25	17	0
do.....	Mine ore.....	4.3	33	0
Colorado.....	do.....	5.5	18	0
Seranton.....	do.....	2.07	18	4
Wilbert.....	Tailings.....	5.11	12	0

The sample from the Horn Silver dump was the only one that contained any noteworthy proportion of lead sulphate; hence the percentage of total lead present as sulphate was not tabulated. In this particular sample the percentage of the lead present as sulphate was 45 per cent. The figures on sulphide lead are based on the proportion of lead insoluble in acidified ammonium acetate solution. According to this criterion a saturated solution of salt, acidified with sulphuric acid, will dissolve considerable quantities of lead sulphide. Small amounts of lead sulphide were present in nearly all the ores tested, although they are classed as carbonate ores.

The amount of this low-grade material available is hard to estimate, because, as a rule, much low-grade ore has been left underground in mining the higher grade ore, but is known to be very large. Each of the tailing dumps mentioned contains many thousands of tons and there are known to be many similar dumps in all of the western base-metal mining States. The amount of low-grade ore left underground in the Tintic district of Utah is very large, as the zone of oxidation extends to a depth of at least 1,500 feet, and is known to be much deeper in many mines.

As the lead carbonate is often so intimately associated with the oxidized gangue minerals that it is practically impossible to separate the particles of lead carbonate from the gangue except by fine grinding, with loss of lead in the slimes, and as many lead carbonate ores refuse to respond to flotation processes that are adapted to slime treatment, hydrometallurgical or pyrometallurgical methods are the only ones that can be applied to many of the carbonate ores. Where sulphidizing and flotation can be applied, it will be of great value in the treatment of these ores, as many of them yield only a small proportion of their gross value to gravity methods of concentration.

METHODS OF TREATMENT.

Hydrometallurgical, pyrometallurgical, and flotation methods have been worked out for the treatment of the low-grade ores, slimes, and tailings, and are included in this report.

The hydrometallurgical method depends upon the fact, previously hinted at, that lead chloride and lead sulphate are soluble in a saturated solution of sodium chloride, and that lead carbonate and lead oxide also can be dissolved if the brine is acidified with either sulphuric or hydrochloric acid.^a Acid-brine solutions could be applied to any material that does not contain too much acid-consuming gangue to prevent practical application of the method. From such solutions it has been found practicable to recover the lead by electrolytic precipitation, as metallic lead, or by the use of lime, as hydroxide of lead. These facts, which seemingly permit a simple process for the recovery of lead from such low-grade ores, form the basis of one of the proposed processes.

If such an ore contains silver in forms other than the chloride the brine solution will not dissolve the silver and some other step will have to be added to the treatment, such as a chloridizing roast to precede the leaching. This point has not been worked out to the same extent that the leaching of the lead has, for the reason that the methods of chloridizing silver are already well known. Only a few tests have been run to see whether the lead and the silver could be leached simultaneously after a chloridizing roast for the silver.

The flotation of argentiferous lead carbonate ores has been extensively tested, and on the ores to which it is adapted the results are encouraging. In order to make the particles of lead carbonate float from a pulp of the ground ore it has been found necessary to form a film of lead sulphide over them by introducing soluble sulphides such as hydrogen sulphide or sodium sulphide into the water in which the ground ore is suspended. The lead carbonate particles, by interaction with this material, are superficially converted into sulphide of lead. These particles can then be caused to enter the froth of a frothing-flotation process. For the ores containing silver in forms that are insoluble in brine, flotation is also a logical procedure. Most of the insoluble silver minerals are sulphides and are especially amenable to flotation. Furthermore, if the lead is only partly oxidized and is partly soluble in brine the natural sulphides of lead might be floated with the artificially filmed particles. Hence, there seems to be a definite field for each of the methods described.

^a For a preliminary discussion of this subject, see *Salt in the metallurgy of lead*, by O. C. Ralston, C. E. Williams, M. J. Udy, and G. J. Holt, *Am. Inst. Min. Eng. Bull.* 125, August, 1917, pp. 1205-16.

SCREEN ANALYSES OF MATERIALS CONTAINING LEAD CARBONATE.

Returning to a consideration of the properties of the materials in question, the results of a number of screen analyses are given in Tables 3, 4, and 5. It is easy to see why the statement is often made that such materials tend to slime too much, because the percentage of lead in the fine sizes is very large compared to that in the coarser sizes. The tailing from the Wilbert dump contains some lead in all the sizes, indicating that the degree of crushing necessary to liberate all the lead has not been attained.

TABLE 3. — *Screen analyses of oxidized lead ores.* ^a
 [Sample crushed to pass 10-mesh standard Tyler screen.]

TAILINGS FROM MAY DAY MILL.

Size.	Percent- age of weight.	Cumulative percent- age of weight.	Lead content.			Silver content.				Silo, per cent.
			Percent.	Weight, grams.	Percent- age of total.	Cumulative percent- age of total.	Ounces per ton.	Weight, mg.	Percent- age of total.	Cumulative percent- age of total.
Through 10-mesh.....	100	100	3.25	3.25	100.00	100.00	2.75	9.48	100.00	100.00
On 20-mesh.....	43.0	43.0	.6	.26	7.97	7.97	2.62	3.86	40.7	40.7
On 35-mesh.....	21.35	64.35	1.9	.40	12.29	20.26	2.50	1.83	19.3	60.0
On 65-mesh.....	13.32	77.67	1.9	.25	7.67	27.93	2.84	1.40	13.7	73.7
On 100-mesh.....	4.98	82.65	11.56	.58	17.80	45.73	2.90	.49	5.2	78.9
On 150-mesh.....	4.45	87.10	9.1	.41	12.60	58.33	3.10	.48	5.1	84.0
On 200-mesh.....	4.90	92.00	8.7	.43	13.20	71.53	3.10	.66	7.0	91.0
Through 200-mesh.....	8.01	100.01	11.56	.93	28.60	100.13	3.24	.86	9.1	99.1

CHIEF CONSOLIDATED MINE ORE.

Size.	Percent- age of weight.	Cumulative percent- age of weight.	Lead content.			Silver content.				Silo, per cent.
			Percent.	Weight, grams.	Percent- age of total.	Cumulative percent- age of total.	Ounces per ton.	Weight, mg.	Percent- age of total.	Cumulative percent- age of total.
Through 10-mesh.....	100	100	2.78	2.78	100.00	100.00	7.85	26.94	100	100
On 20-mesh.....	29.00	29.00	1.90	.55	19.8	19.8	7.22	7.19	26.7	26.7
On 35-mesh.....	25.25	54.25	2.4	.61	22.0	41.8	7.74	6.70	28.8	55.5
On 65-mesh.....	14.94	69.19	3.0	.45	16.2	58.0	4.10	4.10	15.2	70.7
On 100-mesh.....	6.35	75.54	3.5	.22	7.9	65.9	8.28	1.81	6.7	77.4
On 150-mesh.....	6.70	82.24	3.9	.26	9.4	75.3	8.70	2.00	7.4	84.8
On 200-mesh.....	4.88	88.12	4.86	.24	8.6	83.9	8.62	1.44	5.3	90.1
Through 200-mesh.....	12.08	100.20	3.76	.45	16.2	100.1	8.92	3.70	13.8	103.9

^a Analyses by M. J. Udy.

TABLE 4.—*Results of screen analysis of material from Horn Silver dump. a*

Size.	Percent- age of weight.	Cumula- tive per- centage of weight	Zn	Pb	Ag	Fe	Insol- uble
			<i>Per cent.</i>	<i>Per cent.</i>	<i>Oz. per ton.</i>	<i>Per cent.</i>	<i>Per cent.</i>
On 35-mesh.....	1.83	1.83	5.5	1.7	2.50	11.0	68.3
On 48-mesh....	7.75	9.58	5.0	2.1	2.83	1.0	74.2
On 65-mesh....	8.20	17.78	7.0	3.8	3.12	3.8	67.7
On 100-mesh....	11.54	29.32	8.3	4.8	4.41	3.5	65.5
On 150-mesh....	6.84	36.16	8.1	3.7	3.41	3.5	65.0
On 200-mesh..	5.12	41.28	9.4	4.2	5.38	4.4	64.0
Through 200-mesh..	58.22	99.90	6.0	10.5	8.41	5.8	53.8

a Analyses by G. J. Holt.

TABLE 5.—*Results of screen analysis of material from Wilbert dump. a*

Size.	Percent- age of weight.	Cumula- tive per- centage of weight.	Lead (per cent).
On 8-mesh.....	0	0	
On 14-mesh.....	1.32	1.32	4.51
On 20-mesh.....	3.27	4.59	5.28
On 28-mesh.....	8.95	12.64	5.32
On 35-mesh.....	10.47	23.11	5.72
On 48-mesh.....	11.83	34.94	5.42
On 65-mesh.....	14.74	49.68	4.57
On 100-mesh.....	16.29	65.97	5.03
On 150-mesh.....	10.91	76.88	4.63
On 200-mesh.....	10.99	86.97	4.58
Through 200-mesh.....	13.03	100.00	7.41
Total.....	100.00		5.34

a Analyses by C. L. Larson.

LEACHING OF LEAD FROM CARBONATE ORES. ^a

At the time the experimental work on lead carbonate ores was initiated little was known of the solubility of lead chloride or of lead sulphate in strong brines, and hence the first work done was to endeavor to convert the lead of the ore into lead chloride, which is known to be soluble in hot water.

The solubility of lead chloride, as given by Landolt and Börnstein,^b is shown in Table 6. A curve plotted from the figures given in Table 6 is shown in figure 1.

If a cheap method of conversion of lead carbonate into lead chloride can be found, as well as a cheap source of heat for heating water, the number of tons of solution necessary per ton of ore to leach out the lead from most of the ores tabulated would not be large

^a Experiments by C. Y. Pfoutz, C. L. Larson, M. J. Udy, H. C. Neeld, C. E. Sims, G. J. Holt, F. G. Moses, J. F. Cullen, C. E. Williams, and O. C. Ralston.

^b Landolt, H. H., Landolt-Börnstein, physikalische-chemische Tabellen, 1905, 3d ed., p. 564.

TABLE 6.—*Solubility of lead chloride.*

Per cent Pb in solution.	Per cent PbCl ₂ in solution.	Temper- ature, °C.	Per cent Pb in solution.	Per cent PbCl ₂ in solution.	Temper- ature, °C.
0.475	0.637	0.0	1.156	1.550	45.00
.548	.685	8.0	1.550	2.080	65.00
.746	.941	19.95	1.892	2.540	80.00
.767	1.030	25.00	2.382	3.200	100.00

PRELIMINARY TESTS.

With this possibility in view, some of the ores were treated with dilute solutions of hydrochloric acid, followed by washing with hot

water. Only small quantities of ore were treated in these tests, 35 grams being the usual amount. It was found that the lead could be extracted in this way. The next step was to use brine to which sulphuric acid had been added in order to generate hydrochloric acid, although it was felt that this would be a failure, as the sodium sulphate resulting from the reaction of sulphuric acid on sodium chloride should precipitate lead sulphate, if no soluble double salts are formed. However, the brines leached out the lead

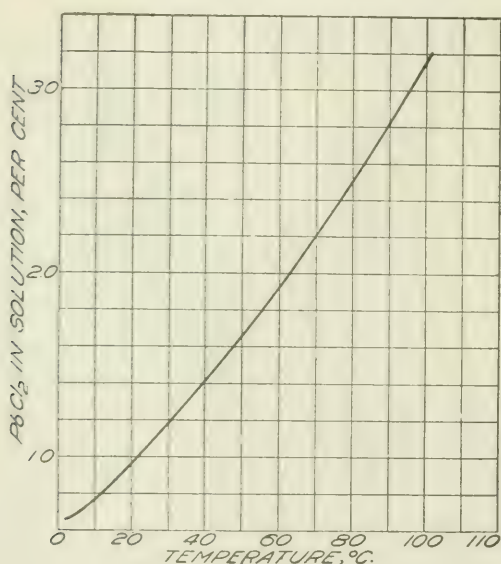


FIGURE 1.—Curve showing solubility of lead chloride (PbCl₂).
After Landolt and Börnstein.

in spite of their containing sulphates in solution. As the final leaching solution to be adopted was a saturated solution of sodium chloride, the results of these tests are briefly summarized in Table 7 following.

The ore treated was from the May Day mine of the Tintic district, Utah, and contained about 5 per cent lead, and some silver.

In the first series of tests shown in this table two variables are involved—the concentration of the acid used, and the amount of acid relative to the weight of ore. The time of contact with the material was of sufficient length to obliterate any effect of the acid concentration. Thus the results of this series of tests show the effect of the relative amount of acid on the extraction of the lead and on the acid efficiency. By **acid efficiency** is meant the amount of acid theoretically needed to convert all of the lead to chloride, compared

to the amount of acid actually consumed. In other words, if one equivalent of lead is extracted by an expenditure of acid equivalent to twice that amount of lead, the acid efficiency is 50 per cent. The acid efficiency was calculated in each test by observing the number of grams of lead extracted, and also the number of grams of acid used up by the ore. One gram of lead theoretically requires 0.3125 gram of HCl to convert it to chloride. If twice that amount of acid was consumed by the ore for every gram of lead extracted, the acid efficiency was $(0.3125 \div 0.6150) \times 100 = 50$ per cent.

TABLE 7.—Results of leaching oxidized lead ore from May Day mine with dilute solutions of hydrochloric acid.

[Tests by C. N. FRANK.]

[Acid applied cold, followed by washing with hot water.]

Sample No.	Time of leaching.	Volume of solution used.	Grams of concentrated acid used.	Acid efficiency.	Lead extracted.
	Hours.	C. c.	Per cent.	Per cent.	Per cent.
A 1	48	100	1.88	30.9	52
A 2	48	100	2.82	33.2	63
A 3	48	100	3.76	36.7	71
B 1	1	100	4.00	15.4	40
B 2	2	100	4.00	26.8	82
B 3	4	100	4.00	28.6	87
B 4	24	100	4.00	23.6	98
B 5	48	100	4.00	45.7	98
C 1	24	100	3.58	23.4	50
C 2	24	150	2.39	11.4	69.7
C 3	24	200	1.79	51.0	79.3

The first series of tests (series A) showed that hardly enough acid had been used. Series B was run with slightly more acid, and the time of contact of the acid with the ore was varied from 1 hour to 48 hours. The results of this series of tests shows that a fairly good extraction can be obtained in a few hours, but that the acid efficiency increases slowly. This latter result is difficult to interpret, as it seems to indicate that after 48 hours more acid is left in the solution than after 24 hours from identical charges. Regeneration of acid is not probable. There is no method of determining accurately the acid content of a solution containing metals such as iron and lead, and it is probable that in the longer tests more of the material that affected the end point in titrating the acid had gone into solution. As much more important and more nearly commercial results were obtained shortly afterwards with the use of brine for leaching, this point was not investigated further.

In the third series of tests, series C, the same weight of acid relative to the weight of ore was used, but was diluted with different amounts of water and the concentration of the acid varied accordingly. Seem-

ingly the more dilute acid converted a greater proportion of the lead to chloride and with a greater efficiency of acid.

The discovery that the brine solutions used in one or more tests were more efficient solvents of lead chloride was coincident with several other lines of evidence learned of at about the same time. The mills of the Holt-Dern Co. in Park City, the Knight-Christensen Co. in Silver City, and of the Bunker Hill and Sullivan Co. in Kellogg, Idaho, were visited. In each of them the use of brine solutions for other purposes had revealed the fact that the lead chloride formed in the particular processes used was soluble in brine solutions to a greater extent than in water. At the Bunker Hill plant it had been

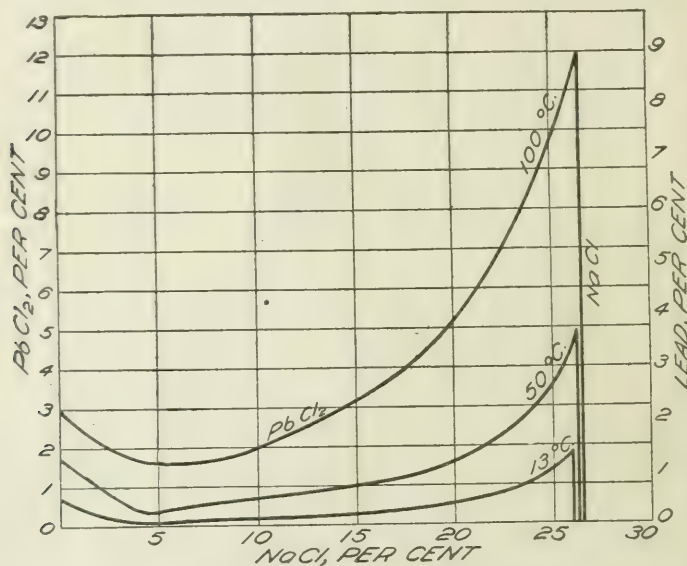


FIGURE 2.—Curves showing solubility of lead chloride in brine.

found that sulphate of lead was likewise soluble in brine. About the same time Demassieux ^a published the results of measurements of the solubility of lead chloride in solutions of salt. His results are given in the curves of figure 2.

The curves show that the solubility of lead chloride in water is at first decreased by small additions of sodium chloride, owing to the well-known "common ion" effect, but that after about 5 per cent of sodium chloride has been added, the solubility of the lead chloride begins to increase within the range of temperatures shown (0° to 100° C.), to the point of saturation of the solution with both PbCl₂ and NaCl, and finally falls to zero again with a solution saturated with NaCl. The increase in solubility in such solutions is usually

^a Demassieux, N., Equilibrium between lead chloride and sodium chloride in aqueous solutions; Chem. Abs., vol. 8, 1914, p. 1899.

ascribed to the formation of a double salt of the two substances involved. No attempt was made to determine the formula of this double salt, as the increased solubility is the important fact in these experiments, the character of the compound formed being chiefly of scientific interest.

The curves in figure 2 show that at any temperature between 0° and 100° C., the solubility of the lead chloride is greatest in the solutions that are more concentrated in sodium chloride. Consequently all brines used as leaching solutions in further experiments were made nearly saturated.

USE OF ACIDIFIED BRINE FOR LEACHING.

FIRST SERIES OF PRELIMINARY TESTS.

To verify the fact that lead chloride was soluble in saturated brine, three different carbonate-of-lead ores were treated with brine to which had been added either sulphuric or hydrochloric acid. Five tests of each ore were made. In the first three tests of each ore increasing amounts of sulphuric acid were added to the brine; the other two tests were with hydrochloric acid. Sulphuric acid reacts with the brine to form hydrochloric acid and sodium sulphate, and it was thought that the sodium sulphate would decrease the solubility of the lead chloride by tending to precipitate insoluble lead sulphate. It was found that sodium sulphate has no such effect unless a considerable proportion is present, and later tests of the solubility of lead sulphate in brines showed that as a rule the addition of sodium sulphate in proportions ranging up to about 2 per cent of the weight of the solution tended to increase the solubility of the lead sulphate. This is a curious chemical anomaly.

As all of the ores tested contained more or less silver, and some of it was known to be present as chloride, the leaches were tested for that metal. The results showed that some silver was being leached.

In each charge 100 grams of 10-mesh ore and 1,000 c. c. of saturated brine (1,210 grams), into which the acid was measured, were used. The charges were agitated in 2.5-liter acid bottles for 24 hours. In the first three tests with each ore the theoretical, three times the theoretical, and five times the theoretical amount of sulphuric acid, respectively, needed to convert the lead carbonate to sulphate were used. In the fourth and the fifth tests the theoretical and five times the theoretical amount of hydrochloric acid needed, respectively, to convert the lead carbonate to lead chloride were used.

The results are shown in Table 8. Excellent extraction of the lead is possible if sufficient acid is applied, but only part of the silver is extracted. The efficiency of the acid is lowest with the ores that contain the largest amount of soluble gangue materials, such as lime,

alumina, and iron. The best acid efficiency obtained with the Chief Consolidated ore was 24.6 per cent, that is, of all the acid consumed only 24.6 per cent did useful work in leaching lead, the rest of the acid being wasted on gangue materials.

As regards the action of hydrochloric acid and that of sulphuric acid, there did not seem to be any great difference except in a few instances. Thus tests Nos. 1 and 4 on the ore from the May Day mine apparently showed that a high extraction of lead was possible with one equivalent of hydrochloric acid, whereas one equivalent of sulphuric acid gave only a low extraction. With five equivalents of acid present the action seemed to be the same. Considerable care was taken in making up the samples, but it is possible that some error crept into one of these determinations, as there is no such marked discrepancy in the other results. Former experiments by Ralston and Gartside^a had shown definitely that there was no difference in the action of the two acids in the leaching of zinc from oxidized ores. Most of the data in Table 8 tend to show the same result for the lead carbonate ores, although there seem to be some minor differences, probably due to errors in the analytical work.

TABLE 8.—Results of leaching lead carbonate ores in acidified brine.

[Tests by M. J. Udy.]

Ore. ^b	Weight of material.		Lead.			Silver.			Acid.			
	Ore in charge.	Tail-ing.	In charge.	In tail-ing.	Ex-trac-tion.	In charge.	In tail-ing.	Ex-trac-tion.	Quan-tity ap-plied.	Quan-tity re-main-ing.	Quan-tity con-sumed.	Acid effi-ciency.
	Gm.	Gm.	Per cent.	Per cent.	Per cent.	Oz. p. ton.	Oz. p. ton.	Per cent.	Gm.	Gm.	Gm.	Per cent.
May Day:												
No. 1 ^b	100	98.2	4.29	3.41	20.5	2.50	2.54		1.99		1.99	20.9
No. 2.....	100	94.0	2.29	.18	96.0		2.40	5	5.97	1.10	4.87	40.1
No. 3.....	100	93.7	4.29	.12	97.4		2.04	23	9.95	5.37	4.58	43.0
No. 4.....	100	97.5	4.29	.63	85.4		2.00	25	1.42		1.42	9.1
No. 5.....	100	93.1	4.29	.10	97.9		2.18	16	7.20	4.87	2.33	63.2
Chief Consolidated:												
No. 1.....	100	97.9	3.10	2.83	8.7	6.88	9.44		1.48		1.48	8.6
No. 2.....	100	93.4	3.10	1.74	33.0		8.02		4.44		4.44	14.5
No. 3.....	100	91.9	3.10	.10	97.0		4.82	30	7.40		7.40	24.6
No. 4.....	100	97.1	3.10	2.42	22.0		5.79	17	1.42		1.42	16.8
No. 5.....	100	89.0	3.10	.20	93.5		3.94	43	7.20		7.20	14.2
Scranton:												
No. 1.....	100	93.5	9.02	5.4	44.0	1.36	1.08	20.6	4.29		4.29	43.9
No. 2.....	100	87.5	9.02	.55	94.6		1.00	26	12.9	4.39	7.51	53.9
No. 3.....	100	89.4	9.02	.98	90.2		1.12	18	21.45	11.45	10.00	39.5
No. 4.....	100	92.0	9.02	5.97	39.2		1.32	3	3.18		3.18	39.1
No. 5.....	100	84.7	9.02	1.26	88.1		1.82		15.90	10.60	5.30	52.5

^a Ralston, O. C., and Gartside, A. E., Leaching a zinc-lime ore with acids: *Met. and Chem. Eng.*, vol. 13, 1915, pp. 151-155.

^b In tests 1, 2, and 3 of each series the amount of sulphuric acid used was, respectively, the theoretical, 3 times the theoretical, and 5 times the theoretical amount; in tests 4 and 5 the amount of hydrochloric acid used was the theoretical and 5 times the theoretical.

SECOND SERIES OF PRELIMINARY TESTS.

As these tests showed that it was possible to leach most of the lead from all three ores tested, and with encouraging consumptions of acid for two of them, a number of other ores from different localities were tested to determine whether they would respond in like manner. The results are shown in Table 9.

TABLE 9.—Results of preliminary tests of leaching lead carbonate ores with acidified brines.

[Tests by M. J. Under, 1,000 c. c. of brine, 100 grams of ore, extended to 10 hours.]

Material tested.	Pb.	Equivalent of acid ^a		H ₂ SO ₄ of acid.	Lead in solution, per cent.	Time of leaching, hours.
		Applied	Consumed			
	Per cent.			Per cent.	Per cent.	Hours.
Ore, Chief Consolidated mine.....	2.81	2.0	2.0	18.2	36.0	16
Do.....	2.81	5.0	5.0	12.7	69.5	16
Do.....	2.81	8.1	7.8	9.5	71.0	16
Do.....	2.81	11.6	11.2	7.3	78.5	16
Ore, American Flag mine.....	3.83	2.0	2.0	17.3	37.3	16
Do.....	3.83	5.0	5.0	12.3	66.5	16
Do.....	3.83	7.0	7.0	10.7	81.9	16
Do.....	3.83	10.0	8.95	10.2	93.0	16
Tailings dump, Wilbert mine.....	5.51	1.0	1.0	63.0	73.0	24
Do.....	5.51	2.0	2.0	42.0	92.0	24
Do.....	5.51	3.0	2.47	31.4	94.6	24
Do.....	5.51	5.0	3.2	27.4	95.2	24
Tailings dump, Horn Silver mine.....	7.16	2.0	1.49	54.5	83.0	16
Do.....	7.16	5.0	1.97	48.6	79.0	16
Do.....	7.16	7.0	1.91	39.0	78.0	16
Do.....	7.16	10.0	3.45	22.2	84.5	16
Do.....	7.16	None.	None.		51.9	16
No. 10 ore, Iron Blossom mine.....	1.94	2.0	.34	5.4	9.7	20
Do.....	1.94	5.0	.32	14.6	19.0	20
Do.....	1.94	7.0	2.14	.7	5.5	20
Do.....	1.94	10.0	2.81	.7	5.6	20
No. 3 ore, Iron Blossom mine.....	3.63	2.0	2.00	14.3	38.6	18
Do.....	3.63	5.0	5.00	7.8	39.4	18
Do.....	3.63	7.0	6.98	2.0	20.2	18
Do.....	3.63	10.0	5.26	7.8	61.5	18
Ore, Daly Judge mine.....	15.33	2.0	1.95	33.0	70.0	20
Do.....	15.33	5.0	2.82	16.9	31.4	20
Do.....	15.33	7.0	4.12	13.9	60.0	20
Do.....	15.33	10.0	5.34	10.2	59.0	20
Ore, Scranton mine.....	8.61	2.0	1.7	53.0	94.0	20
Do.....	8.61	5.0	3.06	29.0	94.0	20
Do.....	8.61	7.0	3.83	23.0	93.8	20
Do.....	8.61	10.0	4.4	20.0	92.5	20

^a 1 equivalent of H₂SO₄ = 474 cental per pound of lead.

In each test a pulp consisting of 1,000 c. c. of brine to 100 grams of ore was prepared. The leaches were agitated all night and on the next day were filtered for analysis. The temperature at which these experiments were run was that of the laboratory, about 20° C. The extractions of lead from the Daly Judge carbonate ore were not as high as was expected from comparison with the figures in Table 2. Later tests showed that it was difficult to get more than 1 per cent of lead into solution in a brine at the laboratory tempera-

tures, hence the use of 10 volumes of solution to 1 weight of ore was slightly below the requirements for the proportion of lead in this ore. Only one type of ore, Iron Blossom No. 10, gave discouragingly low extractions. This ore was really a silver ore containing a small amount of lead. In all of the materials tested the silver content was disregarded for the time being, as the object was to study the lead in as many different ores as possible. As regards acid efficiency the ore from the Scranton mine and the tailings from the Wilbert and the Horn Silver dumps seemed to use less acid per unit of lead

extracted than the other samples, and further tests were therefore made with these materials.

The amount of acid used in each test is expressed as an equivalent to the lead. That is, 1 pound of lead is equivalent to 0.474 pound of sulphuric acid (anhydrous), or, roughly, one-half pound, so that in general 1 pound of sulphuric acid should convert 2 pounds of lead to a soluble form, provided some of the acid is not used up by other constituents of the ore. The data in Table 9 were used in preparing the curves in figure 3, except that the results have been converted to the number of pounds of acid that gave an extraction of a definite

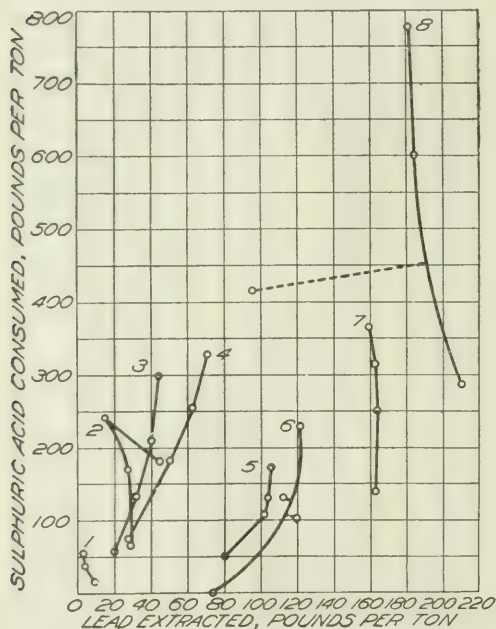


FIGURE 3.—Curves showing relation of lead extraction to consumption of acid in first series of leaching tests with acidified brine. 1, Iron Blossom No. 10 ore; 2, Iron Blossom No. 3 ore; 3, Chief Consolidated ore; 4, American Flag ore; 5, tailing from Wilbert dump; 6, tailing from Horn Silver dump; 7, Scranton ore; 8, Daly Judge ore.

number of pounds of lead from a ton of ore. This is the information that is needed in any calculations as to the financial possibilities of such a process.

From these curves it is evident that with some of the ores the amount of lead extracted after adding more than two equivalents of acid is actually smaller than with less acid. This result is probably due to the excess of sulphate radical present that tends to reprecipitate lead sulphate from the solution. The materials that acted in this way are the ones that did not contain noteworthy amounts of acid-consuming constituents, and hence gave high acid efficiencies.

Down to the limit of the amount of acid necessary to convert the lead carbonate to a soluble form it is best not to use any more acid than such material seems to require. With some of the other materials the addition of more acid always resulted in the extraction of a little more lead.

THIRD SERIES OF PRELIMINARY TESTS.

Another set of argenterous lead carbonate ores was treated in the same way, with the results presented in Table 21 (p. 53), which is incorporated with the section on leaching such ores. The data on the lead extraction from these ores have been calculated and plotted in figure 4, to make the results comparable with those in figure 3. Although the various ores tested vary widely in lead content, it is a strange coincidence that in only a few ores will the use of more than 150 pounds of sulphuric acid per ton of ore give a higher extraction of lead than is obtainable with that amount of acid. In those experiments, however, the main consideration governing the amount of acid used was the possible extraction of the silver in the ore. Therefore the data on the lead extractions are only of technical interest.

TESTS OF TAILING FROM WILBERT DUMP.

Two of the samples that seemed to give the best acid efficiencies were the Wilbert tailing and the Scranton ore. These were chosen for further experimentation. It was necessary to know the minimum time required to extract the lead and the minimum amount of brine that would hold it in solution. It also seemed possible that the application of stronger acid solutions would dissolve the

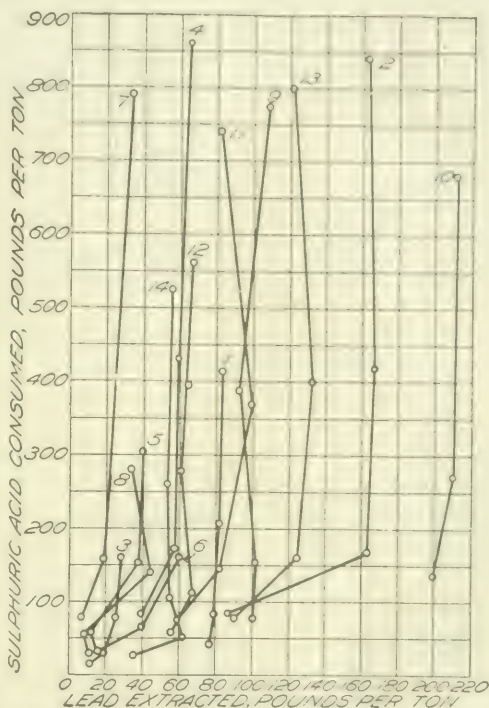


FIGURE 4.—Curves showing relation of lead extraction to consumption of acid in second series of leaching tests with acidified brine. 1, tailing from Ontario dump; 2, tailing from Bullionville dump; slimes from Eureka Hill dump; 4 to 7, Michigan-Utah ores; 8, Chief Consolidated ore; 9, slimes from Copper Queen dump; 10, Shattuck ore; 11, tailing from Dry Valley dump; 12, tailing from Daly West dump; 13, slimes from Bullion Beck dump; 14, Nevada United ore.

lead faster than weaker acid solutions containing the same weight of acid in a greater volume of brine. All of these points were tested on a sample of the Wilbert tailing, of the screen analysis given in Table 5 (p. 19). The charges consisted of 100 grams of ore, usually with 1,000 c. c. of saturated brine unless otherwise stated. The material was placed in ordinary 2.5-liter acid bottles, which were placed on their sides on a cyanide agitator and rolled over about once every two seconds. After agitation the contents were filtered on suction filters consisting of a pump, flask, and a Büchner porcelain funnel.

These data are presented in Table 10. Some of the results are plotted in figure 5. The curves showing the effect of time on the acid efficiency represent two series of tests, one of solutions containing two equivalents of acid, and one of solutions containing four

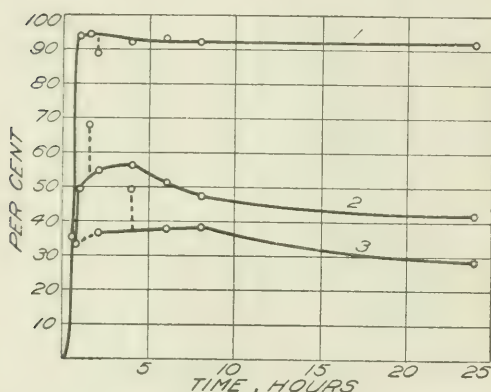


FIGURE 5.—Curve showing effects of time on lead extraction and acid efficiency in leaching Wilbert tailing with acidified brine.

than when two equivalents are used, so both efficiency curves were plotted. The curves show that there is no advantage to be gained by prolonging a leach for more than two hours, either on the score of lead extraction or of acid efficiency. In fact, after two hours both the lead extraction and the acid efficiency fall off to some extent. One would expect that any excess of acid, on long standing, would gradually waste itself on other constituents of the ore; hence, the fall in efficiency of the acid is easy to understand. It may be that on long standing certain other constituents of the ore would also precipitate some lead, as lead is a metal that might be easily precipitated from solution by finely divided iron or particles of other minerals such as zinc oxide or zinc sulphide.

As these tests showed that two hours of leaching was sufficient, it was thought that a series of leaches of varying acidity might reveal the most advantageous concentration of acid to use. However, the percentage of extraction of lead and the acid efficiency seemed to

lead faster than weaker acid solutions containing the same weight of acid in a greater volume of brine. All of these points were tested on a sample of the Wilbert tailing, of the screen analysis given in Table 5 (p. 19). The charges consisted of 100 grams of ore, usually with 1,000 c. c. of saturated brine unless otherwise stated. The material was placed in ordinary 2.5-liter acid bottles, which were placed on their sides on a cyanide agitator and rolled over about once every two seconds. After agitation the contents were filtered on suction filters consisting of a pump, flask, and a Büchner porcelain funnel.

These data are presented in Table 10. Some of the results are plotted in figure 5. The curves showing the effect of time on the acid efficiency represent two series of tests, one of solutions containing two equivalents of acid, and one of solutions containing four equivalents of acid. The curve showing the percentage extraction of the lead represents the series of tests of solutions containing two equivalents of acid. The corresponding curve for the results with solutions containing four equivalents of acid would practically coincide with this curve, and was not plotted. The percentage of efficiency of the acid when four equivalents are used is considerably lower

be unaffected, or at least were not affected in 2-hour leaches. The strength of the acid solution may possibly have some effect on the extraction or on the acid efficiency in leaches of less than two hours' time. This point was not investigated, as the results of the former series of tests, with two of the extremes of acid concentration used, showed that about two hours of contact between the solution and the ore was needed to get the most satisfactory extraction.

TABLE 10.—*Results of leaching Wilbert tailing with acid (test leach).*

(Tests by M. J. Adams.)

Time.	Volume of brine.	Strength of acid solution.		Acid efficiency.	Percentage of lead.	Remarks.
		Concentration.	Acid equivalent.			
<i>Hours.</i>	<i>Cubic centimeters.</i>	<i>Per cent.</i>		<i>Per cent.</i>	<i>Per cent.</i>	
24	1,000	0.442	2.0	42.0	92.7	
8	1,000	.442	2.0	47.5	92.0	
6	1,000	.442	2.0	51.6	94.0	
4	1,000	.442	2.0	57.0	92.0	
2	1,000	.442	2.0	53.5	88.7	
1.5	1,000	.442	2.0	68.0	90.7	
1	1,000	.442	2.0	73.0	90.0	
0.5	1,000	.442	2.0	33.0	75.0	Time of contact too short in short-time leaches.
24	1,000	.884	4.0	28.4	91.0	
8	1,000	.884	4.0	38.2	92.0	
6	1,000	.884	4.0	38.0	93.0	
4	1,000	.884	4.0	49.7	95.0	
2	1,000	.884	4.0	37.2	91.0	
2	1,000	.442	2.0	51.5	88.5	
2	1,000	.552	2.5	53.0	91.0	
2	1,000	.664	3.0	46.5	89.5	
2	1,000	.774	3.5	51.5	91.5	Series to determine effect of strength of acid in short-time leaching.
2	1,000	.884	4.0	37.2	91.0	
2	1,000	.996	4.5	49.0	92.5	
2	1,000	.442	2.0	54.5	88.5	
2	500	.442	2.0	62.0	87.0	
2	700	.442	2.0	48.5	94.0	
2	500	.884	2.0	55.0	92.0	
2	400	1.102	2.0	63.5	91.5	Series to determine minimum amount of brine for use.
2	1,000	.332	1.5	58.0	87.5	
2	900	.369	1.5	59.0	98.0	
2	700	.475	1.5	58.0	98.0	
2	500	.665	1.5	61.0	95.0	
2	400	.830	1.5	58.0	82.0	

The solubility curves for lead chloride in brines (fig. 2, p. 22) indicate that at ordinary room temperatures (about 20° C.), solutions containing as much as 1.5 per cent lead when saturated with respect to lead can be obtained. Such a concentration could not be expected in practice, but it might be possible to get solutions containing considerably more than 1 per cent of lead. Hence a series of tests were run to determine the minimum amount of brine, of a given strength, necessary to leach the lead from the Wilbert tailing, as in the previous tests leaches consisting of 1,000 c. c. of brine to 100 grams of ore that contained only 5.5 per cent of lead had been used.

With complete extraction of the lead such a leaching solution would contain only 0.5 per cent of lead.

Two series of tests with decreasing amounts of brine were tried—one with solutions containing 2 equivalents of acid and the other with solutions containing 1.5. The leaches containing less brine, therefore, had higher concentrations of acid, although the total amount of acid was either 2 or 1.5 equivalents in every test. As the results of the previous series of tests had shown little or no effect due to acid concentration, any effects shown by these two series were probably due to causes other than acid concentration. Leaches were successful down to the point where 400 c.c. of brine to 100 grams of tailing was used, when the lead extraction fell off slightly in the series containing 1.5 equivalents of acid. The acid efficiency was not affected. These solutions contained almost 1.2 per cent lead. Therefore 1 per cent lead was chosen as the probable maximum in practical work at temperatures of about 20° C., and many of the later experiments were so planned that there would always be 1 volume of cold brine for each 1 per cent of lead in the ore. Thus the Wilbert tailing, containing 5.5 per cent lead, could be leached with about 550 c. c. of brine for every 100 grams of ore. As the brine had a specific gravity of about 1.2 at room temperatures this rule will give, roughly, solutions containing somewhat less than 1 per cent of lead. Hence a pulp ratio slightly less than that given by the rule will still have a large factor of safety. In countercurrent decantation it would probably be safe to use pulp ratios that would give solutions more nearly saturated with lead.

TESTS OF MATERIAL FROM SCRANTON MINE.

The results of a series of tests on the Scranton ore, similar to the series on the Wilbert tailing, are recorded in Table 11. Unlike the results with Wilbert tailing, as good an extraction of the lead was made in one-half hour as in 24 hours, and the efficiency of the acid was the same throughout the series. This indicates that practically all of the chemical reactions liable to take place had been completed during the first half hour. As there are few types of modern agitating or leaching machinery that will receive and discharge the ore to be leached in less than a half hour, no tests were made for a shorter length of time. The acid requirements of this ore are such that a solution containing more than 1.5 equivalents of acid must be used before the maximum extraction of lead is possible. The brine requirements of the ore are shown in the third series of tests to be about 700 c. c. of brine to every 100 grams of ore. As the ore contains 8.65 per cent lead, the leaching solutions could be built up to a strength of 1.03 per cent lead under conditions that would give a maximum extraction of lead.

TABLE 11.—*Results of leaching Scranton ore with oxidized brine.*

[Tests by M. J. Hall.]

Time of leaching.	Volume of brine.	Strength of acid solution.		Acid in the clear.	Lead extracted, lbs.	Remarks.
		Concentration.	Acid equivalent.			
<i>Hours.</i>	<i>c. c.</i>	<i>Percent.</i>		<i>Percent.</i>	<i>Percent.</i>	
20	1,000	0.680	2.0	33.0	94.8	
8	1,000	.680	2.0	43.0	92.0	
6	1,000	.680	2.0	48.0	91.0	
4	1,000	.680	2.0	54.0	91.0	
2	1,000	.680	2.0	52.0	90.0	
1	1,000	.680	2.0	54.0	90.0	
1	1,000	.680	2.0	54.0	93.0	
2	1,000	.340	1.0	59.0	82.0	
2	1,000	.51	1.5	63.0	84.0	
2	1,000	.68	2.0	52.0	96.0	
2	1,000	1.02	3.0	48.0	91.0	
2	1,000	1.19	3.5	51.0	90.0	
2	1,000	1.36	4.0	48.0	91.0	
2	1,000	1.53	4.5	49.0	93.0	
2	1,000	.680	2.0	52.0	94.5	
2	900	.775	2.0	48.0	92.0	
2	700	.974	2.0	59.0	92.0	
2	500	1.360	2.0	54.0	89.0	
2	400	1.700	2.0	48.0	79.0	

EFFECT OF SULPHATES IN BRINES.

If any leaching process is employed for removing lead by the use of saturated brine acidified with sulphuric acid, the building up of sodium sulphate in the solution as the result of the action of sulphuric acid on sodium chloride might reach a point where the solubility of the lead chloride in the brine would be seriously affected.

PRELIMINARY TESTS.

In order to determine how much sodium sulphate would do this, a series of rough solubility tests were made in the laboratory of the Bunker Hill & Sullivan Mining & Concentrating Co. at Kellogg, Idaho, by W. J. Winninghoff. The results of these determinations are given in Table 12 following.

TABLE 12.—*Results showing effect of sodium sulphate on solubility of lead sulphate in brine solutions.*

[Tests by W. J. Winninghoff.]

Na ₂ SO ₄ added, grams per 100 c. c. of brine.	Lead in solution at—		Total SO ₄ content (at 21.5° C.).
	96.5° C.	21.5° C.	
	<i>Grams per 100 c. c.</i>	<i>Grams per 100 c. c.</i>	<i>Grams per 100 c. c.</i>
0	4.000	1.405	1.440
1	3.540	1.475	1.435
2	2.590	1.520	1.770
3	2.000	1.750	1.937
4	1.715	1.692	2.065
5	1.495	1.475	2.300
6	1.210	1.115	2.750

The table shows that the solubility of the lead increased with additions of sodium sulphate ranging up to about 3 per cent to brines at ordinary room temperature and then fell off. The maximum solubility of lead in heated solutions (96.5°C.) was in brines uncontaminated with sodium sulphate. Tests at ordinary temperatures by the writers once resulted in a solution containing 1.8 per cent lead, which is very close to Winninghoff's figure, 1.75 per cent lead, when the amount of sulphur in the solution was about 0.646 per cent (equivalent to 1.94 per cent H_2SO_4).

As small proportions of sodium sulphate in the brine do not impair the solubility of the lead compounds, sulphuric acid can be used for acidifying the brine, provided the sodium sulphate is not permitted to build up too high in the solution. All of the various tables of the solubility of sodium sulphate in brines that are available in the literature show that in saturated solutions of NaCl it is possible to dissolve only a small amount of sodium sulphate, and in order to bring any serious amount of sodium sulphate into solution more water must be added. This would handicap the millman by necessitating the use of saturated solutions of NaCl in order to keep the solutions from fouling with sodium sulphate. Most mills using such a solution for treating lead ores would have to precipitate the lead from the solution and use the solution cyclicly, and any building up of sodium sulphate would prove fatal to the process. This objection would not apply to mills on the shores of the Great Salt Lake because the lake brine is nearly saturated with salt, and a constant supply of brine would be available at low cost. At mills situated in isolated localities the tailing could be washed and salt solution recovered, unless the recovered salt proved to be of too little value to pay for the trouble of carrying a wash solution which was gradually being built up to sufficient concentration to be used as leaching solution. These considerations are discussed further in another part of this bulletin.

OTHER TESTS.

To further test the effect of sodium sulphate on the solubility of lead sulphate in the brines, C. L. Larson, in charge of the experimental work at the Bunker Hill and Sullivan mine, performed the following tests. A saturated solution of salt, prepared at a temperature of 17°C. , was found to contain 192 grams of lead per liter and 0.36 gram of sulphur. To this solution was added an excess of PbSO_4 and the whole agitated for 3 hours at a constant temperature of 17°C. The resultant solution contained 171 grams of chlorine, 7.56 grams of sulphur, and 14.95 grams of lead per liter. This test showed that at least part of the lead sulphate had reacted with salt to form lead chloride and sodium sulphate, and that the excess lead chloride

and sodium sulphate were left on the bottom of the container. A similar procedure, carried on at 8° C., gave a resultant solution containing 176 grams of chlorine, 7.35 grams of sulphur, and 13.34 grams of lead per liter. The results of these tests show that the solubility of the lead compounds decreases with decrease of temperature and that the estimate of 1 per cent lead being the proper maximum to use in practical work is probably correct.

Larson found, on treating a solution with excess of sodium sulphate and of lead chloride, that the resultant solutions contained 15.80 and 7.98 grams of lead per liter at 17° and 8° C., respectively. These figures show fair agreement with the results of the tests in which an excess of lead sulphate alone was added, as the excess of lead sulphate probably reacted with sodium chloride to form sodium sulphate. The sodium sulphate undoubtedly lowers the solubility of the lead compounds in the solution, when conditions are such that sodium sulphate is formed and thereby displaces sodium chloride in the solution. Subsequent tests showed that as long as the proper concentration of chlorine in the solution could be maintained the solubility of the lead compounds was not lowered, irrespective of the proportion of sulphur present. In this regard it would be of scientific interest to determine the solubility of the reciprocal salt pair, $\text{PbSO}_4\text{-PbCl}_2\text{-2NaCl-Na}_2\text{SO}_4$.

Several times solutions left out on a cold night lost their sodium sulphate by crystallization at the low temperatures. This happened both in experiments at the Bunker Hill & Sullivan laboratory and in the Bureau of Mines laboratory at Salt Lake City. This illustrates the Höepfner method of removing sodium sulphate by refrigeration, which Höepfner proposed to use in his process for leaching zinc ores. No attempt was made to apply this fact in the experimental work on lead ores, but it suggests a possible method of removing sodium sulphate from the brine solution whenever it builds up to a dangerous proportion. The water of the Great Salt Lake is continually ridding itself of its burden of sodium sulphate in this way. In wintertime this "winter salt" crystallizes out and is driven on shore by the wind, where it quickly dries and is blown away. However, F. G. Cottrell, chief metallurgist of the Bureau of Mines, suggested that the best way to clear the brines of sulphates would be to add calcium chloride to the solution in order to precipitate the sulphur as insoluble calcium sulphate. Calcium chloride is a by-product in a number of the heavy chemical industries and can be bought at a low price or prepared rather cheaply in almost any locality. This step proved to be so easy in manipulation that the problem of refrigeration was not taken up.

CYCLIC LEACHING.

To determine whether solutions would foul in cyclic work, two series of cyclic leaches were designed. In one of them it was proposed to precipitate the lead from solution as a basic hydroxide by the use of lime or milk of lime. In the other it was proposed to recover the lead by electrolysis. Much of the preliminary work in developing the processes that were worked out was on solutions prepared from roasted sulphide ores. A description of those experiments will be found under "Sulphide ores of lead," on pages 125-137. The beginnings of the completed idea were worked out at the experimental plant of the Bunker Hill & Sullivan company at Kellogg, Idaho, by F. G. Cottrell, chief metallurgist of the Bureau of Mines, and R. S. Handy, superintendent of the mill. This plant had been built for testing the Bunker Hill ore with the Malm dry-chlorination process. This process proved not to be adapted for the high-lead, low-zinc ore, but certain chemical facts were noticed that led to proposed processes whose modification in the laboratory at the Salt Lake City station resulted in the process that was ultimately worked out.

LABORATORY LEACHING TESTS.

Material from the Horn Silver tailing dump, Frisco, Utah, was chosen for cyclic leaching tests, as in the solubility tests this material had promised high yields of lead with good acid efficiency, and it contained enough acid-soluble impurities to foul the solution provided the impurities of a tailing, slime, or ore could do so. The sample was a grab sample taken from the surface of the dump.

PROCEDURE AND RESULTS.

Two separate series of small laboratory leaches were made. In one the intent was to precipitate the lead by means of calcium hydroxide; in the other, to precipitate the lead by electrolysis. Later, some larger scale tests were made in which precipitation with lime was used.

The results obtained by precipitation of the lead as hydroxide are shown in Table 13. The work covered 24 complete cycles of leaching a charge of material with brine and precipitation of the lead from the brine with lime.

Too much acid was added to the brine in the first leaches so that the solutions were never quite neutral and the amount of impurities dissolved was excessive. By reducing the amount, of acid used, the lead went into solution better and a much better grade of precipitate was obtained. The lime for precipitation was always slaked in brine in order to prevent dilution of the brine leaching solution.

TABLE 13.—*Results of cyclic leaching of material from Horn Silver tailing dump with acidified brine and precipitation with lime.*
 (Tests by M. J. Uday. Lead content of material, 7.98 per cent.)

Test No.	Solution assay before precipitation.			CaO used for precipitation.	Solution assay after precipitation.			Assay of precipitate.			Tailings.			Ore used.	Solution lost.	Acid used.	Remarks.
	Pb.	S.	CaO.		Pb.	S.	CaO.	Weight.	Pb.	S.	CaO.	Weight.	Pb.				
	C. c.	P. ct.	P. ct.	Grams.	C. c.	P. ct.	P. ct.	Grams.	P. ct.	P. ct.	P. ct.	Grams.	P. ct.	Grams.	C. c.	C. c.	
1	710	0.58	0.865	0.176	821	None	0.352	35	12.20			105	1.75	88.5	200	1,000	Four agitation vessels used in series.
2	600	0.76			880	do.		24	21.22			100	2.04	75	200	1,000	
3	600	0.96										75	1.56	90.2	200	1,000	
4	470	1.06						26	45.70			270	1.80	73	200	1,000	
5	573	1.47						24	32.00			112	1.74	87.8	200	1,000	
6	590	1.43						20	40.80			160	1.75	82.5	200	1,000	
7	560	1.55						17	60.86			177	2.09	76.8	200	1,000	
8	560	1.50						20	51.34			160	2.33	76.7	200	1,000	
9	750	1.80						21	56.55			168	2.04	78.0	200	1,000	
10	950	1.55						26	53.05			155	1.53	84.0	200	1,000	
11	940	1.57						23	57.13			170	2.33	75.1	200	1,000	
12	700	1.46						15	59.16			132	2.33	80.6	200	1,000	
13	710	1.33						15	68.78			160	2.33	77.6	200	1,000	
14																	Fourth vessel removed and acid of glass for 1.
15	930	1.05	.26	.185	3.0	1,000	do.	38	68.79			180	2.33	77.7	200	1,000	2.5 grains of CaCl ₂ added.
16	850	1.19	.408	.219	4.2	950	do.	38	68.79			165	2.91	70.0	200	1,000	
17	870	1.05	.400	.202	4.0	980	do.	345	72.87			180	2.62	71.0	200	1,000	
18	910	.99	.349	.152	3.92	1,000	do.	290	72.87			187	2.33	73.6	200	1,000	
19	600	1.74	.508	.151	5.0	660	do.	37	60.66			157	2.04	82.1	200	1,000	
20	690	1.42	.439	.158	3.0	800	do.	390	72.87			160	2.04	77.0	200	1,000	
21	660	1.54	.411	.107	3.0	750	do.	37	73.31			180	2.04	75.0	200	1,000	
22	850	1.20	.329	.262	2.75	875	do.	329	73.22			175	2.44	73.1	200	1,000	
23	800	1.28	.383	.107	3.26	885	do.	302	65.28			150	2.33	77.8	200	1,000	
24	900	1.17	.383	.190	2.82	990	do.	357	73.46			150	2.04	75.0	200	1,000	

NOTE. Average assay of precipitate in Nos. 14 to 24—Ag, 2.40 oz. per ton; Pb, 70.54 per cent; Zn, 3.15 per cent; S, 1.03 per cent; CaO, 5.42 per cent.

These leaches were applied in a countercurrent decantation system devised by the experimenter, M. J. Udy. A number of leaching vessels were made by cutting off the bottom and lower part of 2.5-liter acid bottles of the ordinary type. The cut bottle, on being inverted, resembled the ordinary Pachuca tank, common in cyanide work. A rubber stopper with a glass tube was placed in the bottle neck; a jet of compressed air through this tube kept the charge of material and solution agitated. No central air lift for agitation, as is commonly seen in Pachuca tanks, was used, but the conical sloping shoulder of the vessel returned to the center all material that tended to settle. Periodically the agitation was stopped, the contents were allowed to settle, and a large proportion of the clear solution was siphoned off. In part of the tests four vessels and in the other tests three vessels were used in series. The thickened pulp was drawn through into the vessel below, while the solution that had been decanted was added to the pulp in the vessel above. In this way nearly barren solution entered the bottom vessel and pregnant solution was decanted from the top vessel. Only fresh material was placed in the top vessel and the material, after passing in turn through each vessel, was finally taken from the bottom one as a thick pulp. This pulp was filtered in a Buechner funnel to recover as much of the brine as possible. New brine to make up for the amount retained in the residue was added from time to time.

DISCUSSION OF RESULTS OF TESTS.

In the material from the Horn silver dump some of the lead is present as sulphide, some as sulphate, and some as carbonate. The determination of the sulphide lead (see Table 2, p. 15) shows that 11 per cent of the total lead is lead sulphide. By determining the percentage of sulphur soluble in ammonium acetate solution and the percentage soluble in water it was possible to estimate the proportion of sulphate lead in the ore, which was about 45 per cent of the total lead content. The proportion of lead that can be recovered as sulphide by use of a flotation machine indicates that the proportion of sulphide lead is nearer 25 per cent than 11 per cent of the total lead content. Evidently the ammonium acetate dissolves some of the sulphide lead in this material. Hence, after it was found that the first leaches contained too much acid, the acid concentration was progressively reduced in successive leaches. The improved grade of the precipitate in the later cycles is evident.

In tests 6, 7, and 8 the amount of lime necessary to precipitate all the lead was added in two portions to determine whether a very rich precipitate could be obtained in one of these fractional precipitations. The results were not encouraging and the method of adding at one time all the lime needed was again resorted to.

Some solution and some pulp were lost by splashing out of the agitation jars; hence the extractions were calculated from the assay of the residue rather than from the assay of the solutions.

In tests 15 to 24 the solutions and the precipitate were assayed for Pb, CaO, and S, and the residue was assayed for lead. In tests 1 to 14 four agitation vessels in series were used. In tests 15 to 17 the fourth vessel was removed as its use seemed to be unnecessary. In fact, all of the acid was used up in the first vessel and the other vessels were merely recovering lead sulphate and giving counter-current washing of the material.

In test 18 the lead content in the solution had fallen to 0.99 per cent, so a fourth bottle was added again. This brought the lead content of the solution up to 1.74 per cent immediately, although about the same extraction was being maintained. A considerable amount of water evaporated during the tests, the amount varying with the time of agitation.

It might be expected that when lime is used all of the sodium sulphate would react with lime to form calcium sulphate, which would accompany the precipitate of lead hydroxide. Calcium sulphate has a definite solubility and hence all of the sulphur is not precipitated in this way. However, it was noticed that whenever lime was added in small excess of the amount theoretically needed, the precipitate contained much more sulphur, presumably as calcium sulphate. In test 21 the excess of lime was cut out, with the result that the grade of the precipitate was improved. In test 22 an excess of lime was again added. The grade of the precipitate was 10 per cent lower, whereas the sulphur content of the precipitate increased again. Solubility relations are also such that a considerable proportion of either the calcium sulphate or the sodium sulphate must be thrown down during leaching. Hence although one might expect an equivalent of sodium sulphate in the pregnant solution for every molecule of lead removed, this condition was usually not realized. Likewise, if all the sulphate sulphur left in solution were to react with the lime to form calcium sulphate, one chemical equivalent of CaSO_4 should be precipitated for every equivalent of lead hydroxide, and the highest grade of precipitate in the combined mixture of Pb(OH)_2 and CaSO_4 would contain 55 per cent lead. It is possible to obtain a precipitate containing as much as 80 per cent lead in leaching material that does not contain large amounts of impurities, although the highest grade of precipitate obtained from this particular series of leaches was 73.46 per cent lead. This result was due to leaving most of the sodium sulphate and most of the calcium sulphate in the ore, although calcium sulphate is soluble in saturated brine to some extent (0.572 parts CaSO_4 per 100 c. c. at 26°C .) and tends to precipitate to some extent on addition of lime.

CONCLUSIONS FROM LABORATORY TESTS.

The general conclusions that are to be drawn from this series of leaches are as follows:

1. It is possible to recover about 80 per cent of the lead in the material tested in the form of a hydrate precipitate containing 70 per cent lead. That is, from each ton of material 128 pounds of lead can be extracted, giving about 175 pounds of precipitate.

2. About 12 to 20 pounds of sulphuric acid and 30 pounds of lime would be required to precipitate the lead from a ton of this material.

3. About one-fifth of the solution was lost in the small-scale tests, or one ton of solution to one ton of material treated. The loss could doubtless be made much less in large-scale work with modern filters and agitating machinery. One ton of solution to one ton of material is about the density of the pulp discharged from a Dorr thickener. With an Oliver filter the proportion of solution left in the tailing ought to be about 20 per cent of the weight of the original material. As the solution would consist of 26.8 per cent NaCl by weight, the salt loss in the tailing would be about 5 per cent of the weight of the original material, or 100 pounds per ton when the pulp was not washed on the filter in order to recover salt.

4. The material in this tailing dump, being already ground to fine consistence, would not require grinding for treatment by the leaching process.

5. Sulphates do not build up in the solution to an appreciable extent, nor are they found to any extent in the precipitate. Hence they largely remain in the material being treated. If the tailing were given a final wash in order to recover brine, the sodium sulphate would be taken back into solution. This fact would seem to make washing of the tailing inadvisable.

6. In case sulphates do build up in the solution to any extent, work on other ores has shown that the sulphate can be precipitated by adding calcium chloride to the solution.

7. Excess of lime is to be avoided as the excess lime not only contaminates the precipitate but carries down more of the sulphate sulphur (in the form of calcium sulphate) that would otherwise stay in solution.

LARGE-SCALE CYCLIC LEACHING.

The results of the experiments just described were so encouraging that steps were taken to interest the company that was then trying to work the material of the Horn Silver dump by flotation. The zinc being present as sulphide, the company was having some success in floating that mineral, together with a small amount of sulphide of lead and some sulphide of silver. Most of the lead and a small

amount of silver were left in the tailing from this plant, and the problem of recovering the lead had not been solved. This company, known as the Caldo Mining Co., sent F. G. Moses to cooperate with the bureau in some large-scale tests.

PLANT USED AND METHOD OF OPERATION.

In these tests a plant of the general form shown in figure 6 was used, each charge of material that entered the system weighing 25 to 50 pounds. This amount was large enough to permit estimating the mechanical difficulties in handling the various products, and also to permit conducting the tests under practically the same conditions as in practice, although the operations tended to be intermittent, whereas similar machinery on a commercial scale could be used continuously. A more representative sample of the dump was taken than the one used in the small-scale tests, in order that the results would have greater value. In each test the brine was siphoned from a storage tank at the head of the plant into the Pachuca agitator. Then the compressed air for agitation was turned on, and the calculated amount of sulphuric acid was added. The charge of material, after being softened with a little brine, was then added, and the mixture was agitated for 2 to 3 hours to permit complete solution of the soluble lead. The raw material, as received, contained a large proportion of moisture and therefore enough solid salt was added to saturate this water, in order that the leaching solution would not be diluted.

It was found that practically all of the lead went into solution in the first 15 minutes. The charge was then drained into a settling tank, allowed to settle, the clear solution decanted to the precipitation tanks, and the thickened pulp filtered on a suction filter. Usually the settling period was overnight, although most of the material settled in 2 to 3 hours. The filter (fig. 7), was a "home-made" filter of the intermittent type, which made it necessary

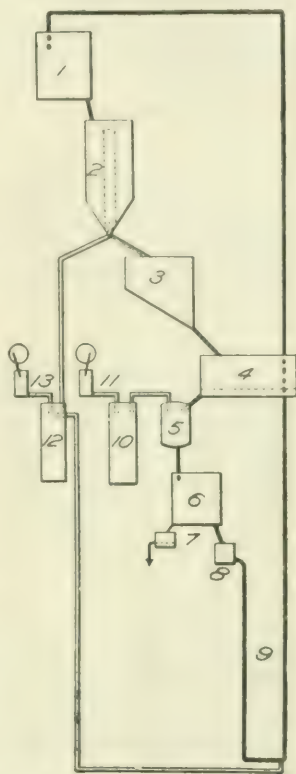


FIGURE 6.—Flow-sheet of test plant. 1, iron storage tank, 4 by 3 feet; 2, iron Pachuca tank, 2.5 by 6 feet; 3, iron settling tank, 4 by 4 by 4 feet; 4, suction filter, 3.5 by 3.5 by 1 foot; 5, solution receiver; 6, precipitation tank, 3 by 2 feet; 7, precipitate filter; 8, sump; 9, air lift; 10, vacuum receiver; 11, vacuum pump; 12, compressed-air receiver; 13, air compressor. All iron parts protected with acid-proof paint.

to operate the rest of the plant intermittently, with batch charges. This made the working of the plant somewhat different from the proposed commercial process, but permitted complete weighing and measuring of all the products formed during a cycle. The filtering period consumed 5 to 6 hours, owing to the imperfect

filter, and the other steps of the process were dependent on the filtering, as it was the slowest step.

The filtered and decanted pregnant solution was treated with the proper proportion of slacked lime to precipitate the lead, the precipitate allowed to settle, and the clarified barren brine siphoned off with a hose, while the thickened sludge was filtered on a smaller suction filter. The barren brine was returned by air lift through an iron pipe to the solution storage at the head of the system.

A compressor and a suction pump operated the plant.

As some sulphuric acid would be used in the flotation of the zinc sulphide (sphalerite) from the material

being treated it was not known how much acid would be left to extract the lead, so a series of preliminary tests of small charges were run to determine how much acid was necessary. It was found that about 15 per cent of the lead was soluble in brine alone. The original material contained 6.5 per cent lead. By the use of about 60 pounds of sulphuric acid per ton of ore, a 72 per cent extraction was possible, leaving 1.84 per cent lead in the tailing, supposedly as galena, as it was insoluble in the acid brine. The results of these preliminary tests are shown in Table 14.

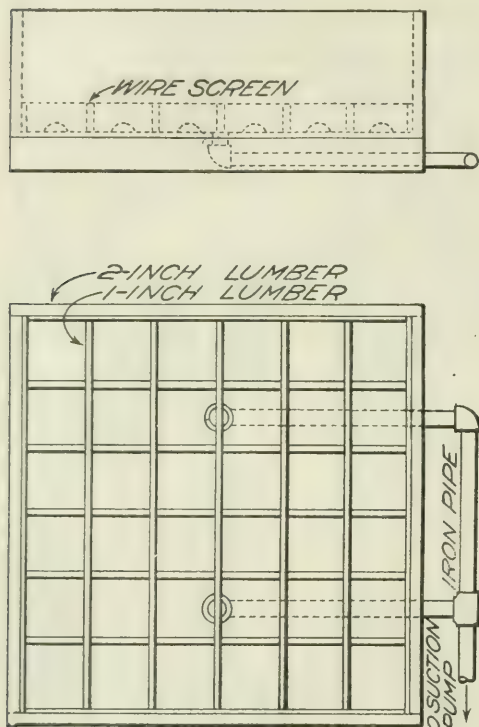


FIGURE 7.—Sketch of laboratory suction filter.

TABLE 14.—*Results of preliminary tests to determine proportion of acid needed.*

[Tests by G. J. Holt and F. G. Moses.]

Test No.	Material used.	Brine used.	NaCl used.	Time agitated.	Lead in residue.	Lead per liter.	Brine waste.	Lead recovery.	Lead recovered per ton of material treated	Acid used per ton.
	<i>C. c.</i>	<i>C. c.</i>	<i>Grams.</i>	<i>Hours.</i>	<i>Per cent.</i>	<i>Grams.</i>	<i>C. c.</i>	<i>Per cent.</i>	<i>Pounds.</i>	<i>Pounds.</i>
1	100	1,000	345	2	5.94	1.37	500	10.44	24.00	62.6
2	100	700	175	16	5.41	1.34	10	18.00	14.20	47.4
3	100	700	175	2	5.90	1.07	280	10.75	95.40	31.3
4	100	700	175	16	1.84	3.81	340	72.20	25.00	15.8
5	100	700	175	16	1.97	4.92	250	70.30	84.20	50.0
6	100	700	175	16	5.36	1.58	120	18.00	74.60	30.0
7	100	700	175	16	5.97	1.92	120	11.20	62.60	10.0
8	100	700	175	16	2.40	5.45	180	65.50	89.20	50.0
9	100	700	175	16	2.58	5.72	125	56.40	84.20	50.0
10	100	700	175	16	2.58	6.07	80	56.40	74.60	30.0
11	100	700	175	16	3.48	4.88	130	47.40	62.60	10.0
12	100	700	175	16	4.88	2.90	80	26.18	34.00	50.0
13	100	700	175	.5		4.58	110		67.80	30.0
14	100	700	175	.5	3.22	4.13	160	51.20	79.20	50.0
15	100	700	175	1	2.65	5.09	20	59.90	81.40	30.0
16	100	700	175	1	2.54	5.50	140	61.50	84.20	50.0
17	100	700	175	2	2.40	6.15		63.70	84.20	50.0
18	100	700	175	2	2.40	5.85	60	63.70	89.20	50.0
19	100	700	175	3	2.15	5.88	120	67.50	83.40	30.0
20	100	700	175	3	2.44	5.05	150	63.90	89.80	50.0
21	100	1,000	250	3	2.12	4.67	70	68.00	89.80	50.0

RESULTS OF TEST AND DISCUSSION OF RESULTS.

The results of the large-scale leaches are recorded in Table 15. In each leach 36 pounds of material, computed on the dry basis from the moisture sample, was used. The material itself was not dried, being taken from the barrel in which it had been shipped in the wet condition. On that account the weight of wet ore is also recorded. Table 16 shows the complete assays that were made on the various products in each of the tests.

TABLE 15.—*General results of large-scale tests of cyclic leaching.*

[Tests by G. J. Holt and F. G. Moses.]

No.	Material used, dry basis.	As received.	Brine used.	Length of agitation.	Lead in tailing.	Lead in solution, pounds per ton of solution.	Lead in barren solution, pounds per ton of solution.
	<i>Pounds.</i>	<i>Pounds.</i>	<i>Pounds.</i>	<i>Hours.</i>	<i>Per cent.</i>		
1	36	48	360	3	2.12	6.46	
2	36	48	360	3	2.54	6.04	
3	36	48	360	2	4.88	4.18	
4	36	48	360	2	2.51	7.40	(a)
5	36	48	360	3	3.11	7.92	(b)
6	36	48	180				Trace.
7	36	48	216	3	2.12	9.68	
8	36	48	298	2	2.43	9.84	1.12
9	36	46	234	3.5	2.59	13.46	2.08
10	36	46	216	2	3.00	11.76	1.75
11	36	46	216	3	2.83	11.08	

a Sample lost.

b Test discarded.

TABLE 15.—General results of large-scale tests of cyclic leaching—Continued.

No.	Lime used for precipitation.	Lead in precipitate.	Quantity of sulphuric acid used per ton of material treated.	Remarks.
	<i>Pounds.</i>	<i>Per cent.</i>	<i>Pounds.</i>	
1	3.40	18.80	50	Excess of lime in solution.
2	1.50	25.60	30	Do.
3	.75	20.80	50	Do.
4	.50	47.51	50	
5	(a)	34.10	50	Lime slaked in water in ball mill.
6			50	
7	.875	21.71	50	Richer solution for precipitation used.
8	.875	21.77	50	Do.
9	.900	35.07	50	Do.
10	.375	41.29	30	Do.
11	.375	26.34	50	Do.

a Test discarded.

TABLE 16.—Results of assays of precipitate, tailing, and solution from cyclic leaches.

[Tests by G. J. Holt and F. G. Moses.]

Test No.	Precipitate.							Tailing.						
	Pb.	Zn.	Fe.	CaO.	S.	Al ₂ O ₃ .	Ag.	Pb.	Zn.	Fe.	CaO.	S.	Al ₂ O ₃ .	Ag.
	<i>P. ct.</i>	<i>P. ct.</i>	<i>P. ct.</i>	<i>P. ct.</i>	<i>P. ct.</i>	<i>P. ct.</i>	<i>Oz. p. ton.</i>	<i>P. ct.</i>	<i>P. ct.</i>	<i>P. ct.</i>	<i>P. ct.</i>	<i>P. ct.</i>	<i>P. ct.</i>	<i>Oz. p. ton.</i>
1	18.8		2.47	25.8	3.93	2.17	7.24	2.12	2.87	4.35	1.88	4.07	6.63	2.64
2	25.6	4.46	1.73	7.74	3.20	.71	7.94	2.54	3.20	4.25	1.41	4.00	6.38	2.76
3	20.8	8.36	1.80	8.48	3.90	1.97		4.88	3.36	5.25	.72	4.16	6.58	2.70
4	47.51	6.73	2.47	8.23	3.55	2.34	13.74	2.51	3.12	3.76	2.48	4.82	4.47	2.90
5	34.1	9.81	2.27	8.48	3.60	.65	23.60	3.11	3.02	4.00	3.12	3.66	6.50	3.12
6	32.9	8.06	3.26	6.64	4.68	4.34	20.70	(a)						
7	21.7	5.60	2.96	16.47	6.90	3.06	10.84	2.12	2.82	4.15	2.40	4.66	7.57	3.00
8	21.77	7.56	2.03	14.90	4.96	.64		2.43	3.51	4.00	2.68	3.84	6.07	3.34
9	35.7		2.22	11.70	5.48	.57		2.59	3.17	4.50	2.87	4.06	7.00	3.80
10	41.3		2.47	6.08	5.76			3.00	2.87	4.49	2.73	4.31	6.23	3.88
11	26.3		7.66	12.30	5.72			2.83		4.25	2.65			

Test No.	Rich solution.				Barren solution.				Lime used for precipitation.
	Pb.	Zn.	Fe.	S.	Pb.	Zn.	Fe.	S.	
	<i>Gm. per liter.</i>	<i>Gm. per liter.</i>	<i>Gm. per liter.</i>	<i>Gm. per liter.</i>	<i>Gm. per liter.</i>	<i>Gm. per liter.</i>	<i>Gm. per liter.</i>	<i>Gm. per liter.</i>	<i>Pounds.</i>
1	3.23	0.73	0.27	0.03			0.01	0.02	3.4
2	3.34	1.19	.21	.03		0.079	.05	.024	1.5
3	2.09	.96	.21	.032		.036	.08	.028	.75
4	4.20	.94	.25	.032	0.42	.826	.23	.032	.50
5	963.96	1.12	1.77	.036	(b)	(b)	(b)		.50
6	(a)	(a)	(a)	(a)	(a)	(a)	(a)	(a)	
7	4.84	.94	.57	.046			.06	.020	.875
8	4.92	2.11	.47	.049	.56	1.190	.03	.030	.875
9	6.73	2.76	.58	.280	1.04	1.820	.02	.030	.50
10	5.88	2.33	.35	.220	.88	.990	.02	.030	.375
11	5.54								.375

a Test discarded because of mechanical trouble.

b Sample lost.

The results were disappointing in two respects—(1) the precipitate was of very low grade as compared with that obtained in the smaller leaches in which all the products could be handled more efficiently; (2), the extractions of lead were rather low.

PURIFYING THE PRECIPITATE.

The first attempts to remedy the low grade of precipitate were made during the large-scale tests, thinking that the trouble was due to the difficulty of mixing the lime with the solution as efficiently on the larger scale as in the smaller laboratory tests.*

PRECIPITATION WITH LIME.

In test 1 (Tables 15 and 16), 3.4 pounds of lime assaying 54 per cent CaO and about 40 per cent MgO was used to precipitate the lead in the solution. A precipitate containing 18.8 per cent Pb and 25 per cent CaO was formed. This precipitate was very dark when fresh but turned white on exposure to air. In the second test the quantity of lime was reduced to 1.5 pounds. This lowered the lime content in the precipitate to 7.74 per cent but raised the lead content to only 25.6 per cent. A considerable amount of sodium chloride was left in this precipitate. In test 3 the lime was reduced to 0.75 pound, giving a precipitate containing 8.36 per cent CaO and 20.8 per cent Pb. The solution after precipitation was barren. In test 4 the lime was cut to 0.5 pound; the grade of the precipitate was 47.5 per cent Pb, which was encouraging. Therefore only 0.5 pound of lime was used in test 5, but the precipitate contained only 34.1 per cent Pb. In test 6 mechanical troubles arose, so that the figures obtained are of no importance.

In these six tests the lime had been slaked in a bucketful of brine and stirred with a stick. In the subsequent tests the lime was slaked in a ball mill in order to break up all lumps and obtain a smooth paste. Also less leaching solution was used in order to make a richer solution for precipitation, the hope being entertained that the grade of the precipitate would also rise under these conditions. In test 7 about half of the amount of solution used in former tests and 0.875 pound of lime were used. The precipitate assayed only 21.71 per cent Pb and 16.47 per cent CaO. The barren solution still contained some lead and it was thought that the reaction had been incomplete. Test 8 was made under similar conditions and more care was exercised, but the resulting product was of about the same grade. In test 9 the lime was cut to 0.5 pound again but the precipitate still contained 11.7 per cent CaO, although it ran 35.11 per cent Pb. However, the solution still contained 1.04 grams Pb per liter after precipitation. In test 10 the lime was further cut to 0.375 pound, with the result that the precipitate assayed 41.3 per cent Pb and 6.08 per cent CaO, but the solution still contained 0.88 gram Pb per liter. An attempt to duplicate these results in test 11 gave a precipitate with 12.5 per cent CaO and 26.3 per cent Pb.

These experiences are very similar to those met with by the Bunker Hill & Sullivan Co. during one stage of its tests. It seemed that too many substances enter the precipitate to permit preparing a product desirable for direct reduction to lead. Calcining the precipitate and leaching with hydrochloric acid failed to remove the lime and other contaminating elements. Enough salt was present to volatilize much of the lead when the precipitate was calcined at a red heat in an effort to raise its grade.

PRECIPITATION ON SCRAP IRON.

This led to an attempt to develop other precipitants for the lead. Scrap iron was tried in an effort to prepare sponge lead. Some of the first iron used was slightly galvanized and the zinc caused quick precipitation of an excellent grade of lead sponge. However, as soon as the zinc was gone, or if pure iron or cast iron was used, the lead did not precipitate.

PRECIPITATION WITH SODIUM SULPHIDE AND HYDROGEN SULPHIDE.

Sodium sulphide and hydrogen sulphide were tested as precipitants. The precipitates from both of these settled slowly and were difficult to filter. Very often the orange-colored sulphochloride of lead was thrown down and the grade of the precipitate was only about 35 per cent Pb. On roasting this material part of the lead was volatilized and the grade of the residue was raised to only 42 per cent Pb.

PRECIPITATION WITH SODIUM CARBONATE.

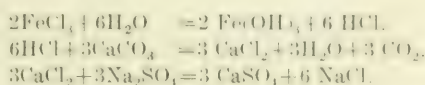
Sodium carbonate gave quick precipitation and a precipitate containing 69.89 per cent Pb, which was very easy to filter, was obtained. On calcining at 800° C. the grade was raised to 77.14 per cent Pb. It was thought that it might be possible to utilize crude "trona" from the alkaline lakes of Nevada and California, but on learning that it would cost nearly 1 cent per pound when delivered in Utah, further search for a cheap precipitant was deemed necessary.

EFFECT OF USING LIMESTONE.

In nearly every test the solution, when ready for precipitation, was slightly acid, and it was thought best to neutralize it with limestone in place of using slaked lime. Ground limestone is used in purifying some of the zinc sulphate solutions in modern electrolytic zinc work, the result being that most of the iron, aluminum, and other metal compounds capable of quick hydrolysis at ordinary temperatures are precipitated. The brine solutions of lead were likewise purified, also the calcium chloride resulting from the hydrolysis precipitated most of the sulphates still in solution. After purification

of the solution in this way, the addition of either slaked lime, sodium carbonate, sodium sulphide, or hydrogen sulphide to the solution resulted in a precipitate containing about 70 or more per cent lead. Roasting the precipitate raised the grade to between 75 and 80 per cent Pb.

The reactions involved in the purification are probably as follows, iron chloride being taken as one of the representative impurities precipitated:



The precipitation results in these tests were checked a number of times with freshly prepared solutions and with solutions that had become foul from cyclic leaching with practically identical results. In a few tests calcium chloride was added to the foul solution; calcium sulphate was precipitated. This precipitate was tested for lead and was usually found to be barren, or nearly so, hence apprehension of losses from inclusion of lead in the calcium sulphate formed during purification of the solution was set aside. Adding the limestone to the pulp before filtration left the calcium sulphate and the hydrolyzed iron and aluminum hydroxides in the tailing, so that only one filtration was necessary. This simplified the process.

TESTS TO DETERMINE BEST CONDITIONS FOR MAXIMUM EXTRACTION.

EFFECT OF VOLUME OF BRINE USED.

After the troubles in precipitation had been solved by finding how to purify the solutions, the question of obtaining the maximum extraction by discovering the best conditions for solution of the lead was next attacked. A series of tests was designed to see what effect different volumes of brine in relation to the weight of the ore would have. It was thought that an excess of brine would be a better solvent than a minimum of brine. As solutions containing not more than 15 grams of lead per liter of solution can be prepared at ordinary temperatures, and it was thought best not to proportion the brine to the ore in a ratio that would give a solution too near the saturation point, a number of ratios calculated to give weaker solutions were used in these tests. The results of the tests are recorded in Table 17. The best results were obtained with the lowest ratio of solution to ore, ratios ranging from 3.25 : 1 to 20 : 1 being tested. The concentration of acid was greatest in the pulp having the solution ratio of 3.25 : 1; hence strength of the acid solution was evidently the determining factor. A method of leaching that would permit the application of all the acid in a very thick pulp, followed by dilution with brine in

order to extract the soluble lead compounds, is indicated. The refractory lead compound that required this special treatment was known to be galena, as all the carbonate of lead and the sulphate of lead dissolve with ease.

TABLE 17.—*Results of tests showing the effects of the ratio of solution to ore on the extraction obtained.*

[Tests by G. J. Holt and F. G. Moses.]

Test No.	Acid used, pounds per ton of ore.	Quantity of material taken for test.	Volume of solution used.	Lead content of tailing.	Lead content of solution, grams per liter.
		Grams.	C. c.	Per cent.	
1	50	100	325	2.13	13.01
2	50	100	500	2.24	8.12
3	50	100	400	2.27	6.72
4	50	100	1,000	2.78	3.52
5	50	100	1,500	2.50	2.95
6	50	100	2,000	2.50	1.75

NOTE.—Material tested assayed 6.77 per cent lead.

EFFECT OF HEATING THE PULP.

Another series of tests were then made to determine whether better extractions could be obtained by heating the pulp. The results, presented in Table 18, show that practically no benefit was derived from heating. The heating of a saturated brine is difficult, because dilution is not permissible. Heating of solutions with exhaust steam would, therefore, necessarily have to be by use of coils of pipe immersed in a tank of brine, which would make the heating expensive. Direct heating with a fire would also be difficult.

TABLE 18.—*Results of tests showing effect of heating pulp, on the extraction of lead.*

[Tests by G. J. Holt and F. G. Moses.]

Test No.	Quantity of material taken for test.	Volume of solution used.	Acid used, pounds per ton of ore.	Time of leaching.	Lead content of raw material.	Lead content of tailing.	Lead content of solution, grams per liter.
	Grams.	C. c.		Hours.	Per cent.	Per cent.	
1	50	250	50	1	6.71	1.55	12.25
2	50	250	30	1	6.71	2.84	9.11
3	100	325	30	1	6.08	2.29	12.53
4	100	325	40	1	6.08	2.29	12.63
5	100	325	50	1	6.08	2.13	13.01
6	100	325	50	1	6.08	2.15	15.45
7	100	325	40	1	6.08	2.29	15.83
8	100	325	30	1	6.08	2.59	14.18
9	100	a 44:1	50	1	6.08	2.13	9.65
10	100	a 44:1	40	1	6.08	2.29	9.65
11	100	a 44:1	30	1	6.08	2.36	8.58

a Ratio of volume of solution (c. c.) to weight (grams) of material tested.

NOTE.—Leaches 1 and 2 were boiled for an hour on the hot plate; Nos. 3, 4, and 5 were leached cold to compare this method with hot leaching; Nos. 6 to 11 were boiled 15 minutes on the hot plate.

EFFECT OF ADDING CHLORIDE OF LIME.

The results of a series of tests to determine whether adding chloride of lime during the leaching would provide chlorine to attack the undissolved lead sulphide are shown in Table 19. It was found that the use of 10 pounds of bleaching powder per ton of material treated increased the extraction of the lead by about 9 pounds per ton of material treated. The price of chloride of lime and that of lead would help determine whether this reagent could be used at a profit. No other methods of increasing the extraction were tested.

TABLE 19.—*Results of tests made to improve leach tailings.*

[Tests by G. L. Holt and F. C. Mose.]

Test No.	Quantity of material taken.	Volume of brine used.	Time treated.	Lead in tailing.	Lead content of solution, grams per liter.	Bleaching powder used, pounds per ton of material tested.	Acid used, pounds per ton of material tested.
	<i>Grams.</i>	<i>C. c.</i>	<i>Hours.</i>	<i>Per cent.</i>			
1	100	500	3	2.09	8.12		50
2	100	600	3	2.22	6.72		50
3	100	1,000	3	2.15	3.52		50
4	100	1,500	3	2.50	2.95		50
5	100	2,000	3	2.39	1.75		50
6	100	400	3	2.82	12.66		50
7	100	300	3	2.02	14.20		50
8	100	500	3	2.03	8.90		75
9	50	250	1	1.55	12.20		50
10	50	200	1	2.89	9.11		30
11	100	350	3	2.29	12.50		30
12	100	350	3	2.29	12.60		40
13	100	350	3	2.13	13.00		50
14	100	450	3	2.12	9.65		50
15	100	450	3	2.29	9.65		40
16	100	450	3	2.36	8.58		30
17	100	400	3	2.75	9.54	10	30
18	100	400	3	1.76	10.50	10	40
19	100	400	3	1.69	10.79	10	50
20	100	400	3	2.40	8.65	20	30
21	100	400	3	1.91	10.13	20	40
22	100	400	3	1.75	10.66	20	50

CONSTRUCTION OF PLANT.

Having determined the conditions under which a plant for leaching lead carbonate ores should operate, it is possible to draw some conclusions as to the commercial application of the process. A proposed flow sheet for such a plant is shown in figure 8. Practically all the machinery would be of standard make and each machine would be of the continuous operating type, these being the only differences between the test plant and the proposed commercial plant. A scrap-iron launder might be used for removing any silver not precipitated with the lead by lime precipitation, as the silver tends to stay in solution. If it should be desired to remove the silver before the lead, the scrap-iron launder can be used ahead of the lime precipitation tank; then the iron passing into solution would be precipitated with

the lead. However, the proportion of this iron in comparison with the lead need not be large, and probably would be negligible. (See "Sulphide ores of lead," pp. 123 to 169.)

MATERIALS USED IN PLANT CONSTRUCTION.

The question of refractory materials for plant construction also received some attention. Iron construction can be used only for barren neutral brine. Acid brine will attack iron, and if the brine contains silver, the silver will be precipitated. On that account Pachuca tanks, thickeners, and filters used for leaching the ore would

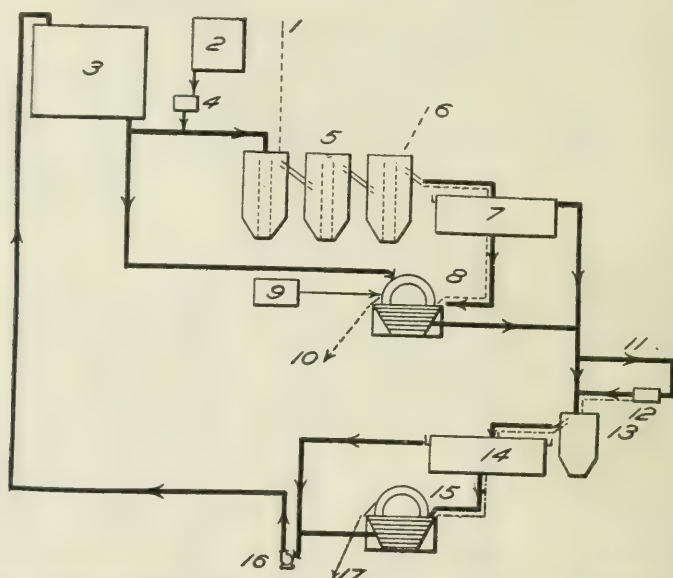


FIGURE 8.—Proposed flow sheet for lead-leaching plant. — solution. - - - - material being treated. precipitate. 1, from dump; 2, acid storage; 3, barren solution storage; 4, acid feeder; 5, wooden Pachuca tanks; 6, ground limestone; 7, Dorr thickener of wooden construction; 8, continuous suction filter (acid-proof); 9, wash water; 10, tailing; 11, brine to slake lime; 12, lime mixer and feeder; 13, precipitation Pachuca tank; 14, Dorr thickener; 15, continuous suction filter; 16, pump to return solution to storage; 17, precipitated basic lead hydroxide.

have to be of wooden construction. Lead pipe used in the construction of the Anaconda Copper Co. copper-leaching plant does not precipitate much silver from the dilute brines of that plant. Hence a lead air lift was used in the Pachuca tank of the testing plant at the Salt Lake City station with seeming success, although the silver extractions were not investigated thoroughly. The ability to use lead in the construction of an acid-proof continuous-suction filter solves that constructional problem, as such filters are already on the market. Also several special irons were tested for their ability to withstand the action of the solutions, and both "duriron" and "corrosiron" were found to be inert toward brines containing silver and lead in solution,

although they are very slightly acted on by the acid. Concrete tanks resist the brine fairly well, and were used for neutral brine in the leaching tests at the Bunker Hill & Sullivan testing plant. Acid brines attack concrete only slowly. Therefore there seems to be no difficulty as to obtaining cheap refractory materials for plant construction. In the testing plant at Salt Lake City some iron tanks, protected with an acid-proof paint were used, but the paint required frequent renewal.

CHARACTER OF SAMPLE TESTED.

Attention should be called to the fact that the sample used for the earlier, small-scale tests was a grab sample of the surface of the old dump, whereas the later large-scale tests were on a representative sample of the tailing from a flotation mill that was treating the material represented by the first sample. The large content of lead sulphate in the first sample was the cause of the low acid consumption in the tests made on it, but the second sample required more acid, for two reasons. The proportion of lead sulphate was less, and 5 to 10 pounds of lime per ton had been used in settling the slime from the tailing water of the flotation plant in order that the water could be used over again. This lime, of course, required an equivalent amount of acid to neutralize it.

CONCLUSIONS FROM RESULTS OF TESTS.

The conclusions drawn by the writers from the results of these experiments are as follows:

1. On a larger quasicommercial scale, the preparation of a solution from which a high-grade precipitate of lead can be thrown down is difficult unless resort is had to purification of the solution with limestone.

2. From the material tested, 83 pounds of lead and 2 ounces of silver per ton could be extracted by the use of 50 pounds sulphuric acid, 20 pounds of lime, with salt losses varying from 20 to 50 pounds, depending on the care used in handling and washing the material and the precipitate.

3. The precipitate obtained with the use of lime will average about 65 per cent lead, or more, and settles and filters rapidly.

4. Sodium carbonate and sodium sulphide give higher grades of precipitate but cost more than lime. The precipitate from the use of sodium sulphide is hard to filter, but the precipitate made by use of sodium carbonate settles rapidly and filters easily.

5. The time required for dissolving the lead need not exceed one-half hour; the time for purification, about one-half hour; and the time for precipitation, 10 minutes. These facts permit the use of small Pachuca tanks for leaching and precipitation.

6. The tailing settles through brine in about three times the time necessary for it to settle in water. Hence ample capacity for thickening the slimes is necessary. Filtering brine likewise requires more filter area than filtering water.

7. The precipitate obtained by using lime settles much faster than the ore being treated and is more easily filtered. Hence much smaller thickeners and filters are satisfactory for such precipitate.

8. The precipitate obtained by using lime may be reduced at the plant or shipped to the smelter direct. Direct reduction of such a high-grade oxidized product of lead is very simple.

CYCLIC LEACHING AND ELECTROLYTIC PRECIPITATION.

ROASTING AND LEACHING TEST AT SALT LAKE STATION.

The second series of laboratory leaches was carried out with electrolytic precipitation instead of precipitation with lime, in order to determine whether the impurities entering the solution under these conditions would detrimentally affect the leaching power of the solutions in any way. Electrolytic precipitation was studied thoroughly before these tests were begun. It was found possible to use iron anodes without affecting the properties of the precipitated lead. The lead always tends to precipitate as sponge lead. This spongy lead will oxidize during drying, and the dried material is readily converted to metallic lead when melted in a reducing atmosphere. Analyses of the dried precipitate revealed as much as 4 per cent of iron in some samples. In melting this material the iron oxide formed a dross, which could be skimmed off, leaving lead of exceptional purity.

The low proportions of lead in the dried precipitate, ranging from 68.4 to 91.7 per cent lead, is due to oxidation of lead and to the presence of small amounts of entrained iron, which later was drossed off. The lead extractions were not determined for all of the tests, as the amount of acid found best in the previous series of leaches (11 pounds per ton) was used. In those determined the extraction was slightly more than 70 per cent. Sixty-two per cent of the silver was extracted by the brine, which accounts for the silver in the final product. The sponge lead could be produced free of silver by precipitating the silver, before electrolysis, on scrap iron, the lead remaining in solution. The silver might also be removed by use of a small amount of the sponge lead, which will slowly displace silver from its solutions. The silver precipitate would form only a small quantity of base bullion when melted. The lead deposited from the desilverized solution would be ready for marketing as soon as melted into bars.

USE OF IRON ANODE.

In this series of tests it was thought that the use of an iron anode, although permitting a very low operating voltage through anodic solution of the iron, might contaminate the solution too much with ferrous salts to permit good leaching of the lead. The first three cycles were run during very cold weather and at night the solutions often cooled nearly to freezing temperature. Whenever this happened, sodium sulphate and other substances crystallized from the solution, so that the content of iron left in the solution after precipitation decreased for a while. The later cycles were run under warmer conditions, and the iron built up to some extent, although a considerable amount must have been left in the ore, from the barren solution at each leach, as the iron left in the solution should be equivalent to the lead removed. Roughly, the iron content of the solution should have increased one-third of 1 per cent on each cycle. The iron was undoubtedly building up during the last tests, as was the sulphur. The material being tested contained some oxidized zinc and it was anticipated that the zinc would likewise build up, but it did not. At the Bunker Hill & Sullivan test plant purifying the solution, after electrolysis, with caustic lime, leaving a precipitate of ferrous and ferric hydrates and calcium sulphate, was tested.

The voltage necessary to obtain a fairly large current density was remarkably low, due, of course, to the use of the soluble iron anode. A poor connection would run up the voltage necessary to operate the cell, as shown by two sets of duplicate conditions in which this effect was studied. The difference in the yield of lead per kilowatt-hour resulting from a slightly higher voltage is very great. All of the data on the solution analyses and the average electrical conditions are given in Table 20. More detailed data on these tests are given in Tables 14 to 16.

TABLE 20. *Results of cyclic leaching and electrolysis of Horn Silver tailings.*

[8 liters saturated brine used per 1,000 grams of material containing 7.4 per cent Pb.]

Test No.	Analysis of solution.					Assay of sponge lead.		Current density, ampere-feet.	Volts.	Current efficiency.	Yield, pounds Pb per kilowatt-hour.
	Before.	After electrolysis.				Pb.	Ag.				
		Pb.	Pb.	Fe.	S.						
	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Oz. p. ton</i>			<i>Per ct.</i>	
1.....	0.778	0.168	1.46	0.71	0.29		18.00	100	1.5	61	2.86
2.....	.785	.026	1.48	.70	.58	68.40	46.17	80	1.3	79	3.43
3.....	.707	.112	1.20	.92	.35	76.90		60	1.0	75	4.66
4.....	1.045	.074	1.17	.93	.65	89.00	103.22	40	.8	90	8.53
5.....	1.080	.085	1.22	1.10	.37	91.70	127.04	40	.7	99	14.30
6.....	.935	.066	1.45	1.33	.58	88.80	63.89	20	.15	95	17.00
7.....	.893	.017	1.68			88.80		20	.5	95	13.72

a Tests by C. E. Sims.

It is remarkable that such high current densities could be employed with a solution so dilute in metal, and still permit of such high current efficiencies as were obtained. The solution being strongly impregnated with sodium chloride accounts for the high electrical conductivity.

TESTS AT BUNKER HILL & SULLIVAN PLANT.

A number of cyclic leaches were made at the Bunker Hill & Sullivan plant with electrolytic precipitation, the cell holding 5 tons of solution. When working on the large scale some difficulty was encountered in washing the brine out of the sponge lead, but compressing the sponge with a hydraulic press remedied this trouble. The cartridges of compressed sponge were in convenient form to place in the melting furnace.

Treatment of the barren brine with lime to remove the iron that had entered the solution during electrolysis was also initiated at this plant, and it was said that no difficulty was experienced in removing the iron. Some calcium sulphate precipitated with the hydrates of iron and made the settling of the precipitate rapid and filtration relatively easy.

More silver was extracted in the tests in which electrolytic precipitation was used than in the series where precipitation with lime was used. This result was believed to be due to the presence of ferric chloride in the leaching solution from electrolytic precipitation. The iron in the solution tended to be reduced at the cathode, so that a preponderance of ferrous iron was present, but there was always some ferric iron present, owing to oxidation by air. The solubility of all compounds of silver in solutions containing ferric iron is well known, and is the basis of many proposed methods for treatment of silver ores, but none have been in recent use.

LEACHING UNROASTED MATERIAL WITH BRINE CONTAINING FERRIC CHLORIDE.

Further work along the lines of leaching the raw ores with brines containing ferric chloride was initiated, but has not been carried to any great extent. A few of the samples at hand were leached with acid brines containing the amounts of iron that were introduced by the electrolysis of the brines in the tests previously described. The electrolyte, after most of the lead has been removed, might be passed through a few cells with carbon anodes in order to oxidize the ferrous iron to the ferric state before being again used as the leaching agent.

These leaches were carried on in parallel with other leaches that were identical, except that no iron was present in the solution. Two series of tests were run. In one the time of contact of solution with ore was 16 hours and in the other series 3 hours. The brines containing

ferrie chloride were so proportioned that 1 per cent of iron, in the ferrie state, was present in the solution. This proportion of iron is rather low, but is only slightly lower than the proportion that was left in the solution after electrolysis. During the leaching with acidified brine the basic minerals of the ore consume the acid and the iron chlorides tend to hydrolyze and be thrown out. Only ferrous chloride survives the leaching operation. The results of these leaches are presented in Table 21. They show that the small amount of iron used caused very little more silver to go into solution than does the use of acid brine alone. Sixteen hours of contact gave lower extractions of the silver than three hours. Most of the materials tested contain some zinc sulphide and other minerals, and the equilibrium conditions would be for the silver to be thrown out of solution and the zinc to pass into solution. Hence it is not surprising that a longer time of contact should give lower extractions of silver.

TABLE 21.—Data on leaching tests made with and without iron in the solution.

[Experiments made by G. J. Holt and C. E. Sims.]

CHARACTER OF LEACHES IN WHICH IRON WAS ADDED TO SOLUTION.

Kind of material tested.	Material taken for test.	Brine.		H ₂ SO ₄ .		FeCl ₃ .	
		Grams.	C. c.	Grams.		Grams.	
Tailing, Horn Silver dump.....	100	780		8		20	
Mine ore, May Day No. 4.....	100	350		5		10	
Mine ore, Chief Consolidated.....	100	350		8		10	
Slimes, Bullion Beck dump.....	100	600		5		18	
Mine ore, American Flag.....	100	500		16		15	
Tailing, Bullionville dump.....	100	1,000		10		20	

RESULTS OF LEACHES.

LEACHES AGITATED 16 HOURS

	Assay.									Extraction.	
	Heading.			Tailing.			Tailing calculated to original weight of ore.			Pb.	Ag.
	Pb.	Ag.	Insoluble.	Pb.	Ag.	Insoluble.	Pb.	Ag.			
With iron:	<i>Per ct.</i>	<i>Oz. per ton.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Oz. per ton.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Oz. per ton.</i>	<i>Per ct.</i>	<i>Per ct.</i>	
Horn Silver.....	7.98	6.20	59.8	2.14	3.92	65.0	1.97	3.6	75.3	42.0	
May Day No. 4..	13.21	3.00	23.1	12.38	3.04	24.5	11.60	2.87	12.2	43.3	
Chief Consolidated.....	2.74	7.58	67.9	.71	3.64	73.0	.66	3.38	76.0	20.0	
Bullion Beck.....	2.36	7.28	67.75	1.19	5.12	73.2	1.10	4.74	53.4	34.9	
American Flag.....	4.52	16.20	72.70								
Bullionville.....	9.41	10.52	72.50	1.31	8.48	85.2	1.11	7.22	88.3	31.3	
Without iron:											
Horn Silver.....	7.98	6.20	59.8	2.14	3.04	64.8	1.97	2.80	95.3	54.8	
May Day No. 4..	13.21	3.00	23.1	13.10	3.08	23.9	12.08	2.98	4.0	.66	
Chief Consolidated.....	2.74	7.58	67.1	.83	8.06	69.75	.80	7.76	70.8		
Bullion Beck.....	2.36	7.28	67.75	1.31	7.24	72.6	1.22	6.70	48.4	8.00	
American Flag.....	4.52	16.20	72.70	.83	17.90	72.6	.88	17.90	81.6		
Bullionville.....	9.41	10.52	72.50	.71	8.28	86.4	.60	6.95	93.6	33.9	

¹ Negative.

TABLE 21.—*Data on leaching tests made with and without iron in the solution—Contd.***RESULTS OF LEACHES—Continued.****LEACHES AGITATED 3 HOURS.**

	Assay.									Extraction.	
	Heading.			Tailing.			Tailing calculated to original weight of ore.			Pb.	Ag.
	Pb.	Ag.	Insoluble.	Pb.	Ag.	Insoluble.	Pb.	Ag.			
With iron:	<i>Per ct.</i>	<i>Oz. per ton.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Oz. per ton.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Oz. per ton.</i>	<i>Per ct.</i>	<i>Per ct.</i>	
Horn Silver.....	7.98	6.20	59.8	2.38	2.30	66.25	2.15	2.08	73.0	66.8	
May Day No. 4..	13.21	3.00	23.1	13.21	2.96	24.7	12.37	2.77	6.4	7.7	
Chief Consolidated.....	2.74	7.48	67.1	.47	3.12	72.7	.43	2.86	84.4	62.2	
Bullion Beck.....	2.36	7.28	57.75	.90	2.28	71.4	.85	2.15	64.0	70.4	
American Flag...	4.52	16.20	72.70	.13	2.48	82.8	.11	2.18	95.6	86.5	
Bullionville.....	9.41	10.52	72.50	.71	8.00	85.1	.60	6.81	93.7	35.2	
Without iron:											
Horn Silver.....	7.98	6.20	59.8	2.38	3.32	67.4	2.12	2.95	73.4	58.3	
May Day No. 4..	13.21	3.00	23.1	13.21	3.00	25.1	12.20	2.76	7.6	8.0	
Chief Consolidated.....	2.74	7.58	67.1	.47	3.82	77.0	.41	3.33	85.0	56.0	
Bullion Beck.....	2.36	7.28	67.75	.90	2.84	74.2	.82	2.59	65.3	64.5	
American Flag...	4.52	16.20	72.70	.13	2.72	82.7	.11	2.39	97.6	85.3	
Bullionville.....	9.41	10.52	72.50	.71	8.30	84.2	.61	7.15	93.6	32.0	

If an oxidized lead ore contains silver in such form that it is easily soluble in acidified brine, there is no need of roasting to make the silver soluble, hence the process of extracting the lead and the silver by leaching would be extremely simple. To test out a considerable range of materials of this type for simultaneous leaching of the lead and the silver, and also any zinc that might tend to dissolve, a series of tests were made on 13 different ores and tailings with varying amounts of sulphuric acid, as calculated for the theoretical requirements of the lead in the material. The results are given in Table 22, which requires little explanation. The amount of acid used is stated in equivalents of the lead and with most of the ores leaches containing 1, 2, 5, and 10 equivalents of acid were used. As some of these materials contained gold, the analyses of the various products for that metal are given. The percentages of gold extraction were not calculated.

Some of the tests in which 10 equivalents of acid were used show lower extractions of lead than those with 5. This is probably due to the large excess of sodium sulphate, formed by action of the sulphuric acid on the sodium chloride. A large excess of sodium sulphate depresses the solubility of the lead compounds in a brine. The large extractions of silver in some of the materials tested indicates that this method could be used to advantage for their beneficiation. With the other materials other methods, as outlined further on in this report, would be more desirable.

TABLE 22.—Results of acid brine leaches of natural oxidized ores.—Continued.

[Tests made by G. J. Holt.]

Source of material.	Equivalent acid content.	Weight of tailing, grams.	Lead.		Zinc.		Silver.		Gold, ounce per ton.
			Per cent.	Grams.	Per cent.	Grams.	Ounces per ton.	Units.	
Ontario mill dump.									
Do.	None a.	a 100	4.38	4.38	2.94	2.94	9.12	912	0.92
Do.	1.	90	.58	.52	2.57	2.51	5.88	588	.01
Do.	2.	87	.48	.42	2.24	1.95	4.90	427	.02
Do.	3.	86	.32	.28	2.40	1.89	4.20	359	.02
Do.	5.	87	.27	.24	2.17	1.89	4.16	352	.025
Bullionville tailings.									
Do.	None a.	a 100	8.88	8.88	1.03	1.03	11.34	1,134	.03
Do.	1.	92	.50	4.51	.61	.55	8.40	772	.02
Do.	2.	84	.90	.76	.51	.43	7.50	650	.01
Do.	5.	89	.64	.57	.42	.37	3.10	270	.035
Do.	10.	86	.85	.73	.32	.28	8.04	690	.02
Eureka Hill mill dump.									
Do.	None a.	a 100	1.69	1.69	1.07	1.07	4.96	496	.08
Do.	1.	97	1.16	1.13	.84	.82	4.50	435	.02
Do.	2.	99	.74	.73	.89	.88	4.52	447	.02
Do.	5.	96	.48	.46	.37	.35	4.28	411	.03
Do.	10.	94	.52	.50	.47	.45	1.96	185	.015
Michigan-Utah No. 1.									
Do.	None a.	a 100	5.92	5.92	2.24	2.24	5.10	510	.02
Do.	1.	96	4.12	3.94	1.68	1.61	7.86	733	.02
Do.	2.	92.5	3.38	3.13	1.54	1.43	6.66	616	.02
Do.	5.	88	2.98	2.98	.31	.33	4.20	420	.02
Do.	10.	84	3.16	2.64	.55	.51	3.68	368	.02
Michigan-Utah No. 2.									
Do.	None a.	a 100	3.22	3.22	4.73	4.73	4.26	426	.00
Do.	1.	99	2.48	2.44	2.72	2.68	2.40	237	.00
Do.	2.	99	2.69	2.65	2.86	2.82	3.03	289	.00
Do.	5.	91	1.48	1.35	2.62	2.36	2.92	286	.00
Do.	10.	91	1.42	1.29	6.00	2.20	1.74	159	.00
Michigan-Utah No. 3.									
Do.	None a.	a 100	3.43	3.43	7.08	7.08	3.96	396	.00
Do.	1.	94.5	2.66	2.66	4.07	3.84	4.60	416	.00
Do.	2.	89	2.80	1.45	3.70	3.70	2.89	289	.00
Do.	5.	84	.53	1.45	87.00	2.87	.96	81	.00
Lost.									
Do.	None a.	a 100	2.45	2.45	2.62	2.62	2.76	276	.15
Do.	1.	97	2.48	2.41	2.29	2.32	6.10	583	.06
Do.	2.	94	2.70	2.51	1.83	1.72	3.58	357	.02
Do.	5.	88	.87	.73	.56	.49	8.70	765	.12
Do.	10.	92.5	1.87	1.27	.60	1.32	8.80	813	.12

a Original material.

TABLE 22.—Results of acid brine leaches of natural oxidized ores—Continued.

Source of material.	Equivalent acid content.	Weight of tailing, grams.	Lead.		Zinc.		Silver.		Gold, ounces per ton.
			Per cent.	Grams.	Per cent.	Grams.	Per cent.	Ounces per ton.	
Copper Queen tailings.	None a.	a 100	8.18	8.18	0.75	0.75		700	0.04
Do.	1.	92	3.48	3.20	.42	.38		564	.00
Do.	2.	89	3.54	3.15	Trace.	0		516	.01
Do.	5.	90	4.01	3.59	Trace.	0		525	.00
Do.	10.	89	3.11	2.77	.14	.12		537	.00
Shatluck.	None a.	a 100	14.40	14.40	.23	.23		1,314	.02
Do.	1.	87	5.17	4.47	.28	.24		1,410	.00
Do.	2.	82	4.75	3.90	.14	.12		1,220	.00
Do.	5.	84	4.54	3.80	.18	.15		Lost.	.02
Michigan-Utah No. 4.	None a.	a 100	8.35	8.35	6.60	6.60		1,150	.00
Do.	1.	95	8.45	8.05	5.38	5.38		810	.00
Do.	2.	95	7.82	1.43	4.40	4.18		627	.00
Do.	5.	86	5.96	5.10	.37	.32		650	.00
Do.	10.	98	6.69	19.90	6.36	6.25		171	.00
Dry Valley tailing dump.	None a.	a 100	7.82	7.82	.61	.61		646	.03
Do.	1.	95	5.23	4.95	Trace.	0		1,080	.00
Do.	2.	90	4.06	3.71	.32	.29		864	.05
Do.	5.	92	3.22	2.90	.14	.13		794	.06
Do.	10.	91	4.17	3.78	.16	.15		637	.05
Daly West tailing dump.	None a.	a 100	5.92	5.92	5.49	5.49		610	.02
Do.	1.	92	3.32	3.04	3.90	3.90		2,020	.02
Do.	2.	89	2.90	2.58	4.26	4.26		1,400	.00
Do.	5.	86	3.43	2.97	3.17	3.17		1,090	.00
Do.	10.	85	3.05	2.59	2.52	2.18		555	.00
Bullion Beck tailing dump.	None a.	a 100	8.45	8.45	2.02	2.22		487	.02
Do.	1.	97	4.16	4.00	1.45	1.45		760	.01
Do.	2.	95	2.38	2.25	1.31	1.26		607	.01
Do.	5.	91	1.86	1.86	.93	.93		600	.00
Do.	10.	90	2.69	2.40	.37	.34		Lost.	.00
					.14	.13		582	.00
								23.40	.00

CONCLUSIONS ON LEACHING AND ELECTROLYTIC PRECIPITATION OF LEAD.

The conclusions to be drawn from the results of the tests of leaching and electrolytic precipitation are as follows:

1. It is possible to precipitate lead from brine by electrolysis, yielding sponge lead that can be melted into pure lead.

2. Impurities do not build up in the solution to an excessive extent and can be removed with lime when necessary. Purification of solutions is not so essential as when lime is used to precipitate the lead.

3. Current efficiencies of more than 90 per cent are possible with current densities ranging up to 40 amperes per square foot of cathode surface.

4. Iron anodes can be used, yielding solutions containing both ferrous and ferric chlorides, and cutting down the voltage to a fraction of one volt. This allows the production of as much as 17 pounds of lead per kilowatt-hour.

5. The presence of ferric iron in the solution permits slightly higher extractions of silver, owing to the ferric iron attacking silver sulphide minerals.

6. Many of the materials tested contained considerable amounts of silver in soluble form.

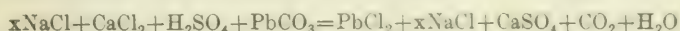
7. Silver can be removed from the solutions by the use of either scrap iron or sponge lead, although the use of iron is preferable because of the greater speed of precipitation. The amount of iron introduced into the solution is negligible.

8. Melting the sponge lead presents few difficulties, although a small proportion of dross is formed. Treatment by careful washing and hydraulic pressing will remove most of the water and most of the salt and will reduce the formation of dross.

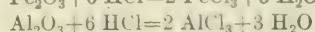
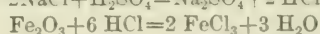
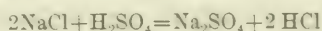
CHEMICAL REACTIONS INVOLVED IN BRINE LEACHING.

In order that the chemistry of the brine leaching processes may be more clearly understood, the following reactions are presented:

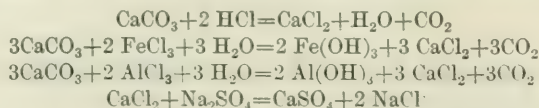
In leaching the chief reaction is as follows:



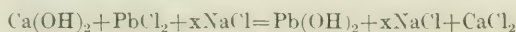
This is the reaction sought as ideal. If not enough calcium chloride is present in the brine returning from precipitation all the sulphates will not be precipitated and some sodium sulphate will be left in the leaching solution. Some of the usual impurities enter the leaching solution by the following reactions:



The purification of the solution is based on the following reactions:



In precipitation with lime the reaction can be written as follows:



For electrolytic precipitation the reaction is—



ROASTING AND LEACHING OF ARGENTIFEROUS OXIDIZED ORES OF LEAD.

In the treatment of lead ores containing silver, the problem of extracting the silver with the lead complicates matters. Unless the silver is present as silver chloride it will usually not dissolve in an acid brine except under the conditions mentioned on page 53, where ferric chloride in the brine seemed to add slightly to its dissolving power, as far as silver is concerned. Hence, some method of chloridizing the silver is needed.

In former days, before the advent of the cyanide process, the principal hydrometallurgical method of recovering silver consisted of a chloridizing roast to render the silver soluble in either a solution of sodium thiosulphate or brine. The latter constituted what was called the Augustine process. Chloridizing roasting was done in the old style reverberatory furnaces, and was acknowledged to be a delicate operation, as there were volatilization losses when the temperature was too high and losses of insoluble silver when other conditions were not just right. In present-day metallurgy losses from volatilization can be prevented with the use of electrical precipitation apparatus, which utilizes high-tension direct-current discharges through gases, but losses of insoluble silver from improper roasting are still to be feared.

TESTS AT ONTARIO MILL.

A year before the experimental work at the Utah station was begun, a modification of the Augustine process was tried in two mills in Utah. One was the old Ontario mill at Park City, treating stope fillings from the Ontario mine. In this mill a Holt-Dern roaster^a was used for the chloridizing roast. The Holt-Dern roaster is a shaft roaster 7 by 9 feet in horizontal section, in which the charge fills the shaft and heat is generated by the burning of fuel mixed with the ore or of sulphides in the ore, a blast of air being supplied from below. Drawings of one form of this roaster are shown in figure 9. The mix-

^a Dern, G. H., and Holt, T. P., Ore roaster: U. S. patent 1113962, Apr. 9, 1914.

ture of ore and fuel is ignited at the bottom and the roasting zone travels upward. The method proposed by the makers involved removal

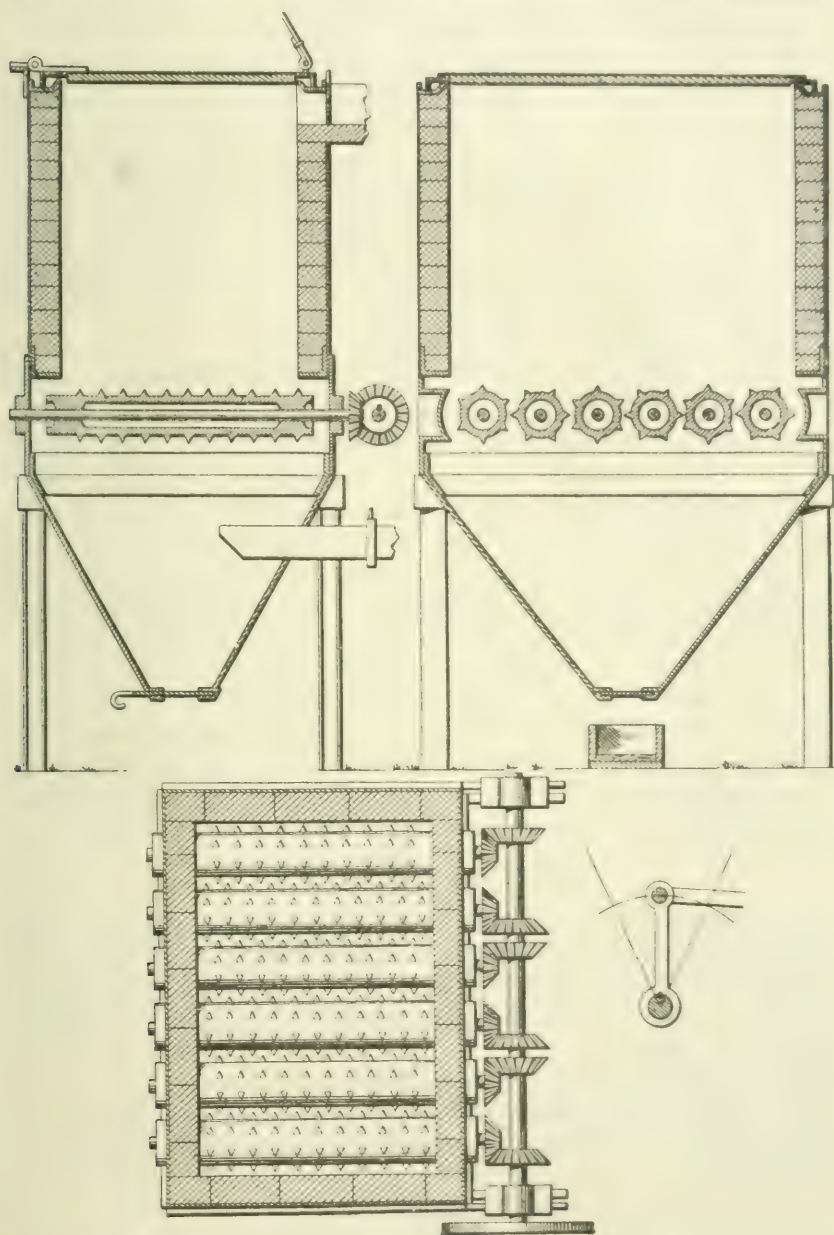


FIGURE 9.—Sections of Holt-Dern roaster.

of the calcines at the bottom at intervals of about an hour, fresh charge being added at the top, so that the roasting zone would be

maintained about midway up the furnace. The cold charge above the roasting zone would catch most of the material that tends to volatilize. The fuel consumption would be low, not more than 2 or 3 per cent of coal dust, or 4 per cent of sulphur being needed.

This roaster was thoroughly tested at the Ontario mill. The results showed that from the oxidized, siliceous material tested an extraction of 90 per cent of the silver, gold, and copper content was possible when a leach containing 20 per cent sodium chloride was used. Such a brine is not a good solvent for lead, although considerable lead was recovered along with the cement copper from the scrap-iron precipitation launders of the mill. The method proposed by the writers involves the use of brine containing 26.8 per cent sodium chloride, which would be a better solvent for the lead.

TESTS AT KNIGHT-CHRISTENSEN MILL.

The other mill in which brine-leaching tests were made was known as the Knight-Christensen mill, in Silver City, Utah, in the Tintic district. The procedure was similar to that at the Ontario mill, except that a down-draft roaster with a shallow bed of ore was used for the chloridizing roast. This roaster, patented by Christensen,^a resembles a circular Dwight-Lloyd sintering machine, with a suction box beneath a porous annular grate. The experimental mill was practically completed when it was burned down. Various parts of the mill had been installed and thoroughly tested and the problem under way at the time it burned was precipitation of the lead, copper, gold, and silver in metallic form, ready for marketing, from the solutions obtained by leaching. The feasibility of extracting all four metals from the ores seemed assured, as the mill leaching solutions had been tested a number of times with favorable results.

BUREAU OF MINES TESTS.

With this much data in hand, tests were made at the Salt Lake City station to determine whether the saturated brines used for the leaching of lead alone could extract silver in the same proportion from an ore that had been properly prepared by roasting in a chloridizing atmosphere.

Three types of material were tested—material from the Colorado mine in the Tintic district, material from the American Flag mine in the Park City district, and tailing from the Wilbert dump in Arco, Idaho.

PROCEDURE IN TESTS.

A Holt-Dern roaster of laboratory size was used in these tests. This roaster (fig. 10) was constructed of reinforced concrete in two sections—a top or shaft and a base. The shaft was filled with a

^a Christensen, N. C., Jr., Method of treating ores and the like: U. S. patent 1075011, Mar. 26, 1913.

charge of ore and fuel which rested on a piece of screen over a wind box in the base. The charge was ignited at the bottom by means of a gas burner, placed in the wind box, that was lighted before the charge was dumped in. As a rule it was also necessary to form a thin bed of glowing coals above the screen before dumping in the charge. After the charge had ignited the gas was turned off, as there was usually no difficulty in keeping up combustion if sufficient air was supplied.

The method of mixing the charge was important. The material had to be mixed dry with powdered coal or coke and then enough water was added to the mixture so that some of it would just hold together when pressed in the hand. When tumbled in a box or passed through a coarse screen, the moistened mass tends to form into little balls about one-fourth inch in diameter. In blastroasting such a mixture will become light and fluffy as it dries out, and most materials will not roast well unless previously prepared in this way. Even a well-recognized "slime" can be balled in this way to permit successful roasting without too great a loss of metal in the dust. Oil may be used instead of solid fuel but must be added after the moisture has been stirred in, as the dry material would absorb the oil so quickly that a few oily lumps would form and the rest of the material would contain no oil.

In the large roasters at Park City material crushed to pass a one-half inch ring was used, but ores that will give good extraction on such coarse crushing are rare. A laboratory roaster will usually require material fine enough to pass through 2-mm. or 1-mm. mesh sieve. The salt (sodium chloride) must be finely granulated in order that good chloridizing roasting can take place. Also, the presence of sulphides of some kind, or of sulphur, is usually required for good chloridizing, as has been mentioned in nearly every textbook on the subject. Pyrite is added to the carbonate ores whenever they

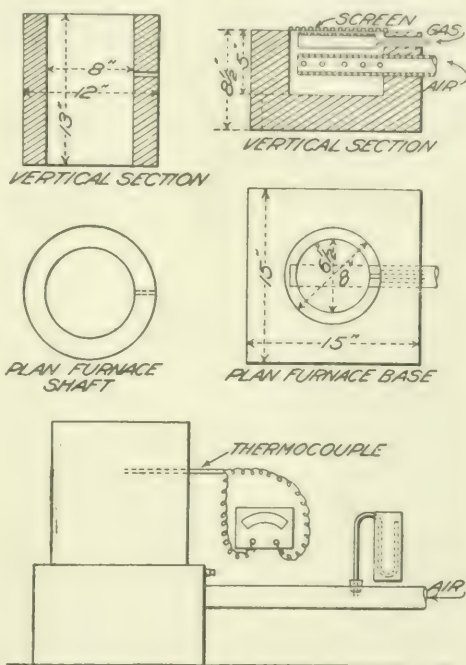


FIGURE 10.—Details of laboratory roaster of Holt-Dern type.

contain no sulphides. The pressure of air applied will determine the temperature of the roast for any given amount of fuel. Temperatures below 600° C. are difficult to maintain. In a laboratory roaster of the size used the roasting lasts one to two hours before fire appears at the top and dies out.

The laboratory roaster had been checked against the large-scale practice in the Ontario mill with somewhat unfavorable results. However, several tests were made with this roaster.

TESTS OF MATERIAL FROM MINE IN TINTIC DISTRICT.

Ore from the Colorado mine in the Tintic district was tested in three different ways. The data of these tests, made by C. L. Larson, are shown in Table 23. Only enough ore for three 20-pound roasts was available.

TABLE 23.—*Results of shaft roasting of argentiferous lead carbonate ore from the Colorado mine.*

[Tests by C. L. Larson.]

Assay of original material: Lead, 5.5 per cent; silver, 7.9 ounces per ton; quantity used per charge, 11 pounds.

Charge.			Volatilized lead, per cent of total.	Percentage of total lead extracted by—			Silver extraction.	Temperature of roast.
Sulphide.	Coal.	Sodium chloride.		Water.	Brine.	Acidified brine.		
<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>					<i>Per cent.</i>	<i>° C.</i>
4.5	1.1	None.	0	0	42	100	48	960
None.	2.3	13.6	4.46	0	11	84	43	625
2.3	1.1	13.6	3.47	12	55	93	70	600

In the first test only pyrite, and no sodium chloride, was added to the ore to determine the effect of the presence of sulphides on the solubility of the lead in the calcines. Supposedly the sulphur in the pyrite would form sulphur dioxide and trioxide and these would react with the lead to form lead sulphite and lead sulphate, both of which are soluble in neutral brine. In order to differentiate between lead oxide or lead carbonate, which is soluble in acidified brine, and lead chloride, which is soluble in hot water, each of the calcines was given three leaches—one with hot water, one with neutral brine, and one with acidified brine. The proportion of sulphate or sulphite lead formed was 42 per cent of the total lead content, and all of the lead was soluble in acidified brine.

In the second test only sodium chloride was added to the ore to determine how efficient it would be in chloridizing the lead without the addition of sulphide to the ore. Seemingly roasting with salt leaves some of the lead in a form insoluble in acidified brine.

The mixture used in the third test, containing both salt and pyrite, fulfills the conditions for good chloridizing of silver as stated by Holt and by Christensen, and mentioned in textbooks on the hydrometal-

lurgy of silver. A fairly good extraction of silver was obtained under these conditions and a 93 per cent extraction of the lead. The results of these three tests hence lead to the conclusion that, under proper conditions for chloridizing the silver in this ore, the lead can be extracted by leaching with acidified brine. Holt found that when he was able to get as high as 70 per cent extraction of the silver in the testing roaster used by him he was usually able to get a somewhat higher extraction in the large roaster, the difference being about 10 per cent. This difference is probably due to the longer length of time the ore was in the large roaster, this time often being 8 to 24 hours.

TESTS OF MATERIAL FROM MINE IN PARK CITY DISTRICT.

Material from the American Flag mine, in the Park City district, Utah, was next tested by the same methods. This material was not much different from that treated by the Ontario mill, except that it was higher in gold. A mill known as the Park City mill was built to treat this material with the intent of using the same kind of machinery and the process used at the Ontario mill. Trouble from the lime content, about 10 per cent CaO in the material finally sent to the mill, and difficult extraction of the gold caused this plant to close down after running one month. To date it has not resumed operations.

Results of the laboratory tests on this material are shown in Table 24. The analysis of the iron sulphide used for aiding chloridization shows 4.4 per cent zinc, 3 per cent lead, and 0.78 ounce silver per ton. The leaches of the unroasted ore and the unroasted sulphide show that practically all of the silver and all of the zinc in both ore and sulphide, and 74 per cent of the lead in the ore and 95 per cent of the lead in the sulphide, were soluble in acidified brine without roasting.

TABLE 24.—*Results of shaft roasting of argentiferous lead carbonate ore from the American Flag mine.*

[Tests by C. L. Larson.]

Assay of original material: Lead, 4.16 per cent; zinc, 6.93 per cent; silver, 15.94 ounces per ton. Assay of sulphide used: Lead, 3.0 per cent; zinc, 4.4 per cent; silver, 0.78 ounce per ton.

Test No.	Charge.				Extraction of—		
	Ore.	Sodium. chloride.	Coal.	Sulphide.	Zinc.	Lead.	Silver.
	Pounds.	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.
1	10	7	5	0.0	50.3	12.0	85.3
2	8	7	3	2.0	71.5	100.0	92.2
3	7	7	2	3.	51.7	100.0	71.3
4	6	7	1	4.0	37.8	100.0	0.0
5	9.5	7	3	0.5	31.3	13.9	44.6
6	9	7	3	1.0	43.3	43.5	62.6
7	8.5	7	3	1.5	58.6	71.6	81.0
8	9.2	5	2	.75	54.1	53.3	87.9
9	9.2	7	3	.75	63.1	60.5	87.9
	Unroasted ore.....				85.9	74.1	89.7
	Unroasted pyrite.....				31.7	95.0	0.0

Average loss of weight in roasting, 7.1 per cent.

The extractions of every metal except lead unexpectedly decreased with the addition of sulphide to the charge before roasting. Adding a small proportion of sulphide seemed to make the lead much less soluble, but increasing proportions up to 40 per cent rendered the lead entirely soluble. With proportions of sulphide of more than 20 per cent the extraction of the silver fell. In every test the zinc in the roasted material was less soluble than in the unroasted material. As a whole, chloridizing roasting failed to improve the extractions of metals from this material and was generally a hindrance to good extraction. It was thought that the lead was converted to basic sulphate to a greater extent in the presence of sulphides during the roasting, and results of later tests confirmed that view. This accounts for the higher extractions of lead, but the causes of the lower extractions of the silver are not known. The fact that the silver in the raw material was soluble would seem to indicate that it was present as chloride of silver, and prolonged heating of silver chloride at the roasting temperatures could cause thermal decomposition of silver chloride into metallic silver and chlorine. Good chloridizing roasting, as mentioned by Hoffmann,^a has always been carried to the point where silver chloride has been formed and no further.

TESTS OF MATERIAL FROM TAILINGS DUMP.

The strange behavior of the lead in these two ores during roasting, led to further experiments to determine the effect of sodium chloride and of sulphide, individually and together, on the roasting of material containing lead carbonate in the shaft roaster. A large supply of the material from the Wilbert tailings dump was at hand and some of it was used for the purpose. The absence of sulphur and of other contaminants made this material desirable for this purpose. Two series of roasts were made, the results are shown in Table 25. In the first series of roasts, common salt was added and no sulphide; in the second series both sulphide and salt were added to the charge, except in two tests in which no salt was used.

Roasting this material at temperatures between 500° and 800° C., with salt added and no sulphide present, did not convert a high percentage of the lead content into chloride of lead. Chloride of lead is soluble in hot water, hence the results of the hot-water leaches indicate the efficiency of the roasting. Unexpectedly, more lead was soluble in hot brine than in hot water after such a roasting, as is shown by tests 1, 2, and 3. However, the efficiency of the process is very low. By using a considerable amount of salt and roasting the mixture twice, in order to give a longer time of treatment, the content of water-soluble lead chloride was increased to 45.9 per cent. As can be seen from the later tests, it was found that the extraction could

^a Hoffmann, Ottokar, *Hydrometallurgy of silver*, 1907, p. 20.

be increased to a satisfactory degree by double roasting of the material after finer grinding, followed by brine leaching. As much as 92.7 per cent of the lead was recovered in this way, nor does it seem possible to get good extractions without such treatment.

TABLE 25.—Results of shaft roasting of material from Wilbert tailings dump.

[Tests by C. L. Larson.]

Assay of original material: Lead, 5.51 per cent; silver, 0.75 ounce per ton.

Test No.	Charge.						Temperature.	Time.	Percentage of total lead soluble in—		Remarks.
	Ore.		NaCl.	FeS ₂ .	Coal.	H ₂ O.			Hot water.	Brine.	
	Fineness, mesh.	Weight.									
		Pounds.	Per cent.	Per cent.	Per cent.	Per cent.	° C.	Hours.			
1	s	25	4.0	None.	2.0	3.5	425	4.0	10.3	15.7	
2	s	23	4.3	do.	3.0	4.0	740	2.0	7.3	18.2	
3	s	22	4.5	do.	2.5	5.0	830	1.5	7.0	19.6	
4	s	22	4.5	do.	2.0	6.0	660	1.75	3.0	8.0	
5	s	20	10.0	do.	2.0	6.5	680	1.3	.0	.0	
6	s	20	5.0	do.	2.5	6.5	810	1.3	45.9		Double roasting.
7	s	20	.0	do.	2.0	7.5	626	1.2			Salt added last, double
8	s		5.0	do.	2.0	6.5	790	1.0	22.0		roast.
9	2s	19	6.5	do.	2.0	6.5	555	1.5	.0		Finely ground, and
10	2s		5.0	do.	2.0	7.2	775	.8	70.8	92.7	double roast.
11	2s	18	5.0	do.	3.0	9.0	790	.7	41.2	60.4	High temperature, and
12	2s		4.5	do.	3.0	10.0	830	.7	67.8	87.4	double roast.
13	100	16	8.0	do.	3.0	9.0	840	1.2	56.8	70.8	
14	s	18	10.0	do.	3.0	8.5	750	1.0	.0		Very fine grinding,
15	s		.0	do.	2.5	8.0	840	1.1	39.6		double roast, with all
16	s	18	3.0	do.	2.0	8.5	640	.8	.0		salt in first.
17	s	16.5	3.0	do.	2.5	7.0	850		12.0		Double roast.
18	s	18	4.0	do.	2.0	8.5	630	.9	1.6		Double roast, cooled in
19	s	17	3.0	do.	2.5	8.0	810	.8	64.6		neutral atmosphere
20	s	18	5.0	do.	3.0	8.5	750	1.0	40.0		(air), quenched in wa-
21	s	18	4.0	do.	2.0	8.0	620	1.0			ter.
22	s		3.0	do.	2.5	8.0	850	1.0	a 27.4 b 49.2		Double roast.
23	s	18	3.0	5.0	.0	6.5	440			85.1	
24	s	18	5.0	7.5	2.0	8.0	865	1.3	1.0	76.6	
25	s	18	3.0	10.0		7.0	758	1.0		64.0	
26	s	18	6.0	12.0		7.0	830	.66		49.3	
27	s	18	None.	12.0	None.	7.0	790	1.0		45.0	
28	s	18	3.0	4.0	2.0	8.0	800	.9		45.2	
29	s	18	None.	5.0	1.0	9.0	640	1.3		32.5	
30	s	18	3.0	5.0	1.0	9.0	700	.75		49.4	
31	s	18	3.0	6.0	.0	8.0	600	.75		41.9	
32	s	18	10.0	6.0	1.0	8.0	700	.8		75.7	
33	s	18	10.0	8.0	.0	8.0	600	.75		78.5	
34	s	18	10.0	8.0	.0	8.0	700	.9		81.8	
35	s	18	10.0	8.0	.0	15.0	645	1.0		79.1	
36	s	18	11.0	8.0	1.5	8.0	820	.8		82.2	
37	s	18	6.6	4.0	2.0	9.0	740	1.0		68.0	
38	100	18	6.6	4.0	2.0	9.0	740	1.2		79.0	Artificial ore, PbO.
39	s	18	12.8	8.0	1.0	9.0	800	1.0		81.2	

a Cooled in air.

b Quenched in water.

The anomalous behavior of a number of the roasts led to the belief that the lead chloride was being converted to some other form not soluble in water. In every test, more lead was soluble in brine than in hot water, although in many of the tests it seemed that the

percentage of insoluble lead was larger than it should be. This might be explained on the assumption that either silicates of lead were forming or some oxychloride of lead. Some of the quick high-temperature roasts that were not in contact with air as long as some of the others gave higher extractions, so it was thought that possibly the exclusion of air during cooling of the calcine might prevent the formation of such compounds. Covering the furnace during cooling partly excluded the air, and it seems that the extraction was higher. Quenching in water also seemed to considerably improve the process. Due to variations in roasting, it was not possible to be sure that these methods of handling the hot calcines were doing what they seemed to do. Therefore, in test 21 part of the calcine was quenched in water as soon as the roasting was completed, and the rest was covered to exclude air. The quenched calcine gave an extraction of 49 per cent in hot water, whereas the calcine that was cooled slowly with partial exclusion of air only gave 27 per cent extraction. This seemed to prove that the lead chloride was being converted into some insoluble complex compound during cooling.

In any event, double roasting of such low-grade material would not be practicable, so the effect of adding sulphide was next taken up. The results of tests 27 and 29 show that with sulphide alone less lead sulphate is formed than when both pyrite and sodium chloride are used. Whether the pyrite helps in the chloridizing or the sodium chloride helps in the sulphating of the lead is impossible to say, as any lead chloride formed would react with the sodium sulphate as soon as leaching was begun. Hence brine is needed for the leaching, as it will dissolve both lead chloride and lead sulphate. In tests 33 to 39 more salt was used and better extractions were obtained. This would make it appear that the presence of pyrite helps chloridizing. Evidently about 8 per cent of sodium chloride and the same of pyrite are necessary to get higher than an 80 per cent extraction of the lead in this material.

An analysis of some of the tailing showed that about 80 per cent of the silver in the original material was also chloridized and extracted.

The action of the lead in the roasts where no pyrite was added was still puzzling. There are several known oxychlorides of lead that are insoluble,^a and one of them is prepared by heating lead chloride. Some chemically pure lead chloride was heated in a porcelain crucible to a low red heat and first melted down. At red heat it began to give off fumes of chlorine and probably some lead chloride volatilized. A yellow residue resulted, which, on being boiled with water, turned white and was mostly insoluble. This seems to indicate the cause

^a Muir, M. M. P., and Morley, H. F., *Watts' dictionary of chemistry*, vol. 3, 1912, p. 126.

of the trouble. In the highly chloridizing atmosphere afforded by the reaction of the oxides of sulphur from burning pyrite on the sodium chloride, the lead chloride formed could not decompose thermally, whereas in the presence of sodium chloride alone the thermal decomposition of lead chloride gradually took place, especially in a stream of air.

CONCLUSIONS FROM RESULTS OF TESTS.

The conclusions to be drawn from these experiments are as follows:

1. Argentiferous lead carbonate ores in which the lead and silver are not already in soluble forms can be chloridized in a shaft roaster of the type used. The experience of two mills operating for considerable lengths of time has shown that these ores will yield their silver, and experiments at the Bureau of Mines station show the conditions necessary for keeping the lead in soluble form.

2. The addition of both salt and sulphide to the charge is necessary in roasting in order that the lead may be converted to a form soluble in a neutral brine. Lead carbonate and the compounds into which it is converted in roasting are soluble in acid brine, so that the roast need not be controlled with the idea of converting the lead into forms soluble in water or in neutral brines. The requisite conditions for properly chloridizing the silver are the addition of both salt and sulphides to the roast in order to get good chloridizing, and these conditions will not interfere with efficient extraction of the lead.

3. Some lead is volatilized at the temperatures used in roasting, but if the temperature is not allowed to rise above 700°C . the amount volatilized will be small, and the use of an electrical precipitator, as shown in later experiments, will collect all of the material that volatilizes.

VOLATILIZATION OF LEAD FROM OXIDIZED ORES.

Throughout the experiments described in the foregoing pages it was noticed that some lead always volatilized in roasting, especially when a charge of shallow depth was used. Presumably the volatilized lead was lead chloride, as the boiling point of lead chloride is placed at 861°C .^a Before any attempt to utilize this fact had been made by the bureau, it was learned that N. C. Christensen had been utilizing this idea in a proposed new process that he was developing for a mining company at Pioche, Nev. Subsequently a number of tests were made by Christensen at the Bureau of Mines station, with the assistance of M. J. Udy.

^a Landolt, H. M., Landolt-Bornstein physikalisch-chemische Tabellen, 1900, 2nd ed., p. 259.

TESTS WITH CHRISTENSEN ROASTER.

Christensen used a laboratory roaster he had developed for testing ore in the Knight-Christensen mill, at Silver City, that had burned. As previously stated, the commercial Christensen roaster for chloridizing ores is a down-draft roaster with a circular hearth of annular shape and a suction box beneath for creating the draft. A shallow bed of ore is used (4 to 8 inches) and conditions for volatilization of lead chloride are much better, as there was no zone of cold ore to condense volatilized material.

The roaster brought by him to the bureau laboratory is shown in figure 11. This roaster was constructed from a piece of 8-inch pipe with a cap on the lower end. The charge was supported on an iron-wire screen. Air was sucked down through the charge with a Root cycloidal blower, the gases being discharged from the blower

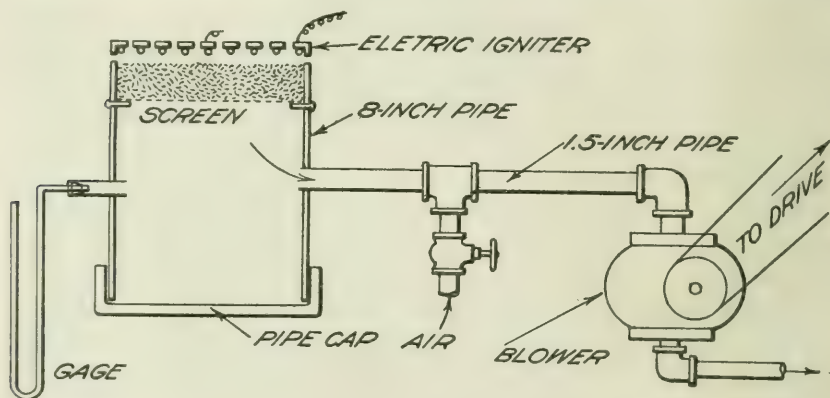


FIGURE 11.—Christensen down-draft laboratory roaster.

on the pressure side. To regulate the draft through the charge a valve in a T joint in the suction pipe was set to cause the proper suction. The charge was ignited by means of an electric igniter made of resistance wire. This igniter consisted of a piece of perforated sheet asbestos on the underside of which was wound a spiral helix of No. 22 B. & S. gage nichrome wire, connected to a 110-volt lighting circuit. The furnace was charged with material that had been crushed and prepared in the same way as for the Holt-Dern laboratory roaster. The igniter was placed over the charge and the current turned on. After the ore on top showed that it was getting hot, by a slight crackling or glowing, the blower was started and immediately the charge would ignite. As soon as the surface of the charge was glowing the current was turned off and the igniter removed. The combustion zone would travel down through the charge in 5 to 20 minutes, and the fire would then die out unless the charge consisted principally of sulphide minerals,

when the whole mass would ignite and burn for as much as an hour. It was found best to support the screen in a smaller piece of pipe that would fit into the pipe shown in the drawing. This piece could be conveniently lifted out with the roasted charge of ore.

The experiments by Christensen with his roaster in the bureau laboratory are described in the following paragraphs. The basic theory was that at higher temperatures than those necessary for chloridizing of silver the lead chloride formed is volatilized. The temperatures used should be near to or above the boiling point of lead chloride, 861°C . Too high a temperature will decompose any silver chloride formed in the roasting process. In roasting a carbonate ore mixed with powdered coal and salt it is a peculiar fact that the only metal to be volatilized as chloride in important amounts at these temperatures and in the length of time allowed for roasting in this furnace is lead. Only a very small amount of silver, gold, or copper volatilizes, and hardly any zinc chloride is formed. In order to get a good extraction of the lead by volatilization, the temperature must be raised so high that all of the silver will not be soluble in saturated brine.

TESTS OF MATERIAL FROM BULLIONVILLE TAILING DUMP.

The first material tested was that from the Bullionville tailing dump near Pioche, Nev. This is finely ground oxidized material containing lead carbonate and some gold and silver. The sample tested had the following content of valuable metals: Lead, 9.7 per cent; copper, 0.32 per cent; silver 10.9 ounces per ton; and gold, 0.135 ounce per ton. The material in this dump had been treated three times previous to being left in its present condition, having been pan-amalgamated twice and cyanided once.

The results of the tests are shown in Table 26. In some of the tests pyritic sulphide was added to the charge to assist chloridizing, and in others limited proportions of sulphide were used and ground coal was added to provide sufficient fuel. In other tests only coal was used in order to determine whether it was possible to chloridize and volatilize the lead without using sulphides. In these tests the stronger the suction the higher was the temperature with a given quantity of fuel.

With increasing temperature more and more of the lead was volatilized. However, in order to leach gold and silver from the tailing with a brine solution, the optimum conditions for their extraction are such that 30 per cent of the lead is not volatilized. The calcines were leached with four parts of brine to one of original material, hence those charges in the tests that show low volatilization of the lead contained too much lead to be taken into solution.

Hence the lead content in the tailings of the first three or four tests was high. However, the conclusion can be made that if the extraction of lead is all that is desired, the lead can be volatilized by mixing the raw material with salt and roasting at a temperature above the boiling point of lead chloride (861° C.) in a blast roaster with a shallow bed.

TABLE 26.—*Results of volatilization of lead chloride from Bullionville tailing.*

[Tests by N. C. Christensen and M. J. Udy.]

Fuel in charge, ^a		Suction, inches of water.	Analysis of calcine.				Analysis of residue from each.			
Sulphide.	Coal.		Au.	Ag.	Cu.	Pb.	Au.	Ag.	Cu.	Pb.
Per cent.	Per cent.		Oz. p. ton.	Oz. p. ton.	Per cent.	Per cent.	Oz. p. ton.	Oz. p. ton.	Per cent.	Per cent.
4		4	0.085	8.6	0.32	8.7	0.05	5.9	0.22	5.7
5		6	.12	9.1	.32	8.7	.04	7.0	.22	4.9
10		6	.125	9.1	.37	8.9	.03	4.9	.22	2.4
10		8	.13	9.45	.37	5.4	.035	4.3		1.4
12		9	.13	9.9	.39	3.55	.035	4.8		1.1
12		12					.04	4.9		.9
4	2.0	12	.10	10.60		2.60	.04	5.6		.6
4	2.5	(?)				1.1				
.....	5.0	(?)				.9				
.....	6.0	(?)				.75				
.....	7.0	(?)				.75				
.....	8.0	(?)				.75				
.....	9.0	(?)				.75				

^a Each roast contained 5 pounds of material, with 7.5 per cent NaCl and proportions of sulphide and coal shown.

TESTS OF MATERIAL FROM DRY VALLEY TAILING DUMP.

To test this conclusion on another material, tailing from the Dry Valley dump, which is in the same mining district as the Bullionville dump, and has been likewise pan-amalgamated, was tested for volatilization of lead and leaching of gold, silver, and copper. The results of an analysis of the material used in these tests was as follows (see also Table 28): Lead, 7.9 per cent; copper, 0.25 per cent; silver, 11.3 ounces per ton; and gold, 0.135 ounce per ton.

TABLE 27.—*Results of volatilization and leaching tests of Dry Valley tailing.*

[Tests by N. C. Christensen and M. J. Udy.]

Fuel in charge, ^a		Suction, inches of water.	Lead content of calcine.	Analysis of leached tailing.			
Sulphide.	Coal.			Au.	Ag.	Pb.	Cu.
Per cent.	Per cent.		Per cent.	Oz. p. ton.	Oz. p. ton.	Per cent.	Per cent.
4	2.5	12	2.0	0.035	4.80	0.6	
4	2.5	12		.02	5.6	.5	0.05
.....				.045	3.1	.5	

^a Each roast contained 5 pounds of material, with 7.5 per cent NaCl and the proportions of sulphide and coal shown.

The fumes in the gases from the furnace were caught in an electrical precipitation apparatus consisting of a 7-foot length of 4-inch pipe set in a vertical position, in the center of which hung an insulated nichrome wire. The pipe was grounded and a pulsating direct current of about 18,000 volts was applied to the wire. The current was derived from a 220-volt alternating-current supply main; it was transformed to a high voltage by use of a variable-voltage transformer and then rectified by a rotating synchronous switch. With velocities of the gases of 10 to 20 feet per second through the pipe, all of the lead chloride in the fumes from the roaster was caught in the electrical precipitator. Testing paper saturated with sodium sulphide solution held in the exit gases failed to blacken except on long exposure, indicating almost complete removal of the lead chloride fume. The composition of the fume caught varied with the position of the sample taken. Some of the fume in the pipe leading from the blower contained 52.5 per cent lead; 0.42 per cent copper; 0.9 ounce of silver per ton, and 0.04 ounce of gold per ton. The fume in the treater itself contained nearly 70 per cent lead. The percentage of lead in pure anhydrous lead chloride is 76.5 per cent. Of course, the dust and the moisture of the charge were caught with the lead chloride, as the gases were well cooled by the time they entered the treater. In larger installations it is possible to so control the cooling of the gases that the temperature is still above the dew point of water when the suspended solids are precipitated, and in this way obtain a dry precipitate.

RECOVERY OF LEAD FROM FUME.

Some question existed as to the recovery of the lead from the lead chloride after it had been volatilized and caught in the precipitator. This was answered by a number of tests performed by H. J. Morgan and M. J. Udy. Lead chloride was mixed with powdered calcium carbonate and with coal in order that the lead might be reduced and the calcium carbonate (or lime, if preferred) converted to calcium chloride, which would melt at 774°C . and form a molten slag over the lead. Such a charge was easily melted and the recovery of lead was high.

A charge consisting of 20 grams of lead chloride, 7.25 grams of calcium carbonate, and 1 gram of powdered coal, was melted at a low red heat in an assay crucible, yielding 14.9 grams of lead or 86 per cent recovery. A similar charge ten times larger, and containing by calculation 150 grams of lead, yielded 130 grams of lead or 87 per cent recovery.

A charge consisting of 50 grams of lead chloride, 50 grams of powdered limestone, and 2 grams of powdered coal yielded 32.4 grams lead or 91.2 per cent recovery. In breaking up the slag to examine it,

shots of lead were noticed; when collected these increased the recovery to 92.4 per cent.

The calcium chloride formed was dark and very hygroscopic. It proved effective as a substitute for sodium chloride in the roasting. Losses of lead in these tests were probably greater than would happen in large-scale practice, as pouring all of the small amount of material from each crucible was difficult, and a relatively large surface area was exposed for volatilization of lead chloride. Any lead volatilized should be caught again in the electrical treater and returned, so that recovery of most of the lead from the lead chloride is practically assured.

A qualitative test to determine the action of zinc chloride mixed with the lead chloride showed that most of the zinc chloride volatilizes and none is reduced.

CONCLUSIONS FROM RESULTS OF TESTS.

The possibilities of recovering lead from its carbonate ores by mixing common salt with the ore and treating the mixture at a high temperature, may be summarized as follows:

1. By mixing common salt and fuel with pulverized lead carbonate ore and heating the mixture in a blast roaster at a temperature near 861°C ., the boiling point of lead chloride, extractions of more than 90 per cent of the lead are possible. The lead is volatilized and carried in the gases as chloride of lead.

2. Chloride of lead can be recovered from the gases by means of an electrical precipitator.

3. The condensed chloride of lead can be reduced to metallic lead by mixing with a small proportion of powdered coal and the proportion of powdered limestone or lime theoretically needed to form calcium chloride with the chlorine, and melting the mixture.

4. The calcium chloride slag is suitable and desirable for use in the roaster as a chloridizing agent in place of sodium chloride.

5. The reduction of cuprous chloride has been accomplished at the plants of the Anaconda Copper Co. and of the Chuquicamata Copper Co. in practically the same way, and on a larger scale, so that the proposed method of reducing lead chloride should be capable of application without inherent difficulty.

6. Many of the materials tested were ground to pass a 0.147-mm. (100-mesh) screen. The coarsest size of particles that will permit extraction of the lead by volatilization has not been determined, although satisfactory extractions were obtained with 0.833-mm. (20-mesh) material.

COMBINED VOLATILIZATION AND LEACHING OF ARGENTIFEROUS LEAD CARBONATE ORES.

As is shown by Tables 17 and 18, page 46, the partial extraction of lead by volatilization at low enough temperature not to destroy any chlorides of silver, gold, and copper which have formed, leaves a product from which these chlorides, with the rest of the lead, can be leached with acidified brine. Therefore, the temperature of the blast roast must be kept from rising much above 800° C., and the material must not be allowed to form a hard sinter.

RESULTS OF EXPERIMENTS AND DISCUSSION OF RESULTS.

From the results obtained by Christensen in his experiments at the bureau laboratory and in the laboratory of the Prince Consolidated Mining Co., amounting in all to about 50 tests, the following figures are summarized for the treatment of the materials from the Bullionville and the Dry Valley tailing dumps, and from the Prince Consolidated Mine:

Summary of extraction data.

	Ag.	Ag.	Pb.
	Oz. p. 100.	Oz. p. 100.	Per cent.
Bullionville:			
Original material.....	0.135	10.9	9.7
Residue.....	.04	4.7	.8
Residue corrected.....	.035	4.2	.7
Extraction.....	.074	.062	a 93
Dry Valley:			
Original material.....	.135	11.3	7.9
Residue.....	.035	3.9	.6
Residue corrected.....	.03	3.5	.55
Extraction.....	.078	.070	a 93
Prince siliceous ore:			
Original material.....	.02	7.0	4.6
Residue.....	.005	1.0	.8
Residue corrected.....	.0045	.9	.7
Extraction.....	.078	.087	a 85
Prince iron ore, from 20-foot bed:			
Original material.....		2.5	3.2
Residue.....		.17	.6
Residue corrected.....		.16	.55
Extraction.....		.094	a 84
Prince iron ore, "big bed":			
Original material.....		2.7	2.9
Residue.....		.02	.5
Residue corrected.....		.29	.45
Extraction.....		.080	a 85
Mixture, one-half Bullionville tailing, one-half Prince iron ore:			
Original material.....	.077	9.0	7.2
Residue.....	.02	3.0	.8
Residue corrected.....	.017	2.7	.7
Extraction.....	.077	.070	a 90
Mixture, one-half Dry Valley tailing, one-half Prince iron ore:			
Original material.....	.077	9.2	6.3
Residue.....	.02	2.6	.8
Residue corrected.....	.017	2.5	.7
Extraction.....	.077	.075	a 89

a Percent.

The iron ore used was from a large deposit in the Prince Consolidated mine at Pioche, Nev. It contains 30 to 35 per cent iron and 10 to 15 per cent manganese. Iron and manganese are supposed

to assist chloridizing of silver, so their presence should not be regarded as detrimental. The extraction of silver from this material is better than from the Bullionville or the Dry Valley tailing. Hence, a mixture of the two materials was thought advisable, but the percentage extractions of silver and gold from the mixture seem to be only the average of the extractions from these materials when individually treated. The presence of manganese dioxide in the acid brine solutions generated considerable free chlorine, but the extraction of gold was not correspondingly increased.

Taken as a whole, the extractions obtained in the small roaster are encouraging, and past experience gives definite assurance that they represent a probable minimum of what larger roasters could do.

CONCLUSIONS AS TO RESULTS OF TESTS.

Summarizing, it is possible to treat argentiferous carbonate ores of lead by blast roasting with common salt, volatilizing 60 to 70 per cent of the total lead as chloride; the rest of the lead and a large proportion of the gold and silver can be extracted from the residue by the use of saturated brine or of acid brine. Total extractions were about 90 per cent of the lead, 60 to 94 per cent of the silver, and 75 per cent of the gold in the materials thus far tested. Iron and manganese seemed to be of some slight advantage in the ore during the chloridizing roast, by making possible higher extractions of silver than can be gotten from the siliceous materials. About 7 per cent of common salt, by weight, of material treated is necessary in this process.

CHLORIDE VOLATILIZATION OF OXIDIZED LEAD, SILVER, GOLD, AND COPPER ORES.

From the foregoing it can be seen that the investigation of methods of treating the oxidized lead ores gradually led from simple leaching to roasting and leaching combined, but that there was always some volatilization of lead. By further raising the temperature of the roast and controlling the atmosphere it was possible to volatilize most of the lead and also most of the gold, silver, and copper. In every instance the chlorides of these metals were volatilized. Volatilization introduced a step toward simplifying the process, as the apparatus proposed for use in some of the earlier tests could be used without the leaching tanks.

HISTORY OF THE PROCESS.

The step involving complete volatilization of all the valuable metals was taken at the suggestion of G. H. Wigton, of the Chief Consolidated Mining Co., who spent some time in the bureau's laboratory

investigating what had been done at the station in treating lead carbonate ores containing some silver and gold. Most of the roasting tests had been in blast roasters, but Wigton used an assayer's muffle. By mixing the ore with common salt and heating to a relatively high temperature, he was able to volatilize not only the lead but also the gold and silver. He attributed this result to the more highly oxidizing atmosphere of the muffle. Several somewhat similar experiments were made by the bureau after being apprised of his success.

Chloridizing roasting has been applied to the treatment of silver ores for many years. The process consists in converting the silver in a nonsmelting ore into silver chloride, dissolving the chloride with a desirable lixiviant, and precipitating and recovering the metal from solution. Recently this practice has been extended to the ores of copper and gold. The work at the Salt Lake station has shown the possibility of treating lead ores in this way. It is well known that this chloridizing roast, when done for leaching only, entails volatilization losses. The temperature is maintained at 700°C . and sometimes rises higher, causing large losses of lead or copper, and comparatively large losses of silver by the old method. The temperature must be kept high in order to effect good chloridization. Therefore, in order to prevent the losses mentioned above, complete volatilization and precipitation by electrical precipitator of the metals is suggested. The complete volatilization process consists in mixing the ore with common salt in the usual way, heating to sufficiently high temperature to drive off the metallic chlorides, precipitating the chlorides, calcining these chlorides, and converting them into the metals by refining.

This method is by no means a new one. It was repeatedly proposed in the earlier days of western metallurgy. A large number of patents^a have been taken out within the past 25 years, in which several different methods of volatilization are covered. The processes proposed in these patents involve the use of the alkaline or alkaline-earth chlorides, and most of them of sulphur or iron pyrites, it being claimed that the presence of sulphur is necessary for chloridization. The Pohlé-Croasdale process was tested commercially, a testing plant of fairly large capacity being operated in 1903 by a metal refining company, of Denver, Colo. The fumes were collected in cooling towers, filtered, and refined. The failure was due chiefly to the inefficient method of catching the volatilized metals, also the tonnage treated seemed to be extremely small for the size of the furnace used.

^a Chanute, Arthur, U. S. patent 501559, Jan. 9, 1892; Besemfelder, E. R., U. S. patent 543284, Dec. 27, 1893; McNight, Robert, U. S. patent 683982, Dec. 5, 1900; Pohlé, E. C., and Croasdale, Stuart, U. S. patent 741712, Jan. 23, 1900; Clawson, S. I., U. S. patent 1192037, Oct. 23, 1912; Forland, T. R., U. S. patent 1078779, Mar. 27, 1913; Clawson, S. I., U. S. patent 1169330, May 24, 1911; Biggs, W. H., English patent 11031, July 29, 1915.

DESCRIPTION OF EXPERIMENTS.

Tests were made at the Salt Lake City station by C. E. Williams, of various oxidized and semioxidized ores of lead and copper that contained gold and silver, and some contained zinc. The roasts were made in a gas-fired assay muffle, small roasting dishes containing 100-gram charges of ore mixed with sodium chloride and other reagents being used. The proper proportions of sodium chloride and calcium chloride, the effect of oxidizing and reducing atmospheres, and the desirability of using iron pyrites, coal, or manganese dioxide, were determined. In all of these tests the fumes were allowed to escape. The results of the tests are shown in Tables 28 to 35, following.

TABLE 28.—Data on tests of material from Nevada United mine, Ely, Nev.

[Analysis of heading: Pb, 5.58 per cent; Ag, 3.20 ounces per ton; Fe, 37.6 per cent; CaO, 1 per cent; Al₂O₃, 2.2 per cent; S, 1.15 per cent; insoluble, 11.6 per cent. Size: Passed 20-mesh sieve. Charge: 100 grams ore, 10 grams water.]

Test No.	NaCl used.	CaCl ₂ used.	Temperature of roast.	Time.	Calcine.			Percentage of extraction.		Remarks.
					Weight.	Pb.	Ag.	Pb.	Ag.	
	Per cent.	Per cent.	° C.	Min-utes.	Gm.	Per cent.	Oz. p. ton.			
1	6	None	850-875	40	85.5	1.16	2.78	63.9	21.4	
2	6	do.	850-875	60	85.5	.68	2.72	87.1	22.9	
3	6	do.	850-875	90	85.5	.62	1.80	88.1	48.8	
4	6	do.	850-875	40	88.0	1.38	2.60	72.6	23.7	
5	6	do.	850-875	60	85.0	.12	2.24	97.0	36.5	
6	6	do.	850-875	90	86.0	.06	2.24	98.9	36.8	
7	6	do.	875	120	84.0	.11	1.25	97.9	65.0	
8	4	do.	900-950	60	85.0	.40	.97	93.4	72.4	
9	6	do.	900-950	60	85.0	.29	1.52	94.4	57.8	
10	6	do.	900-950	60	85.0	.87	.69	83.4	80.7	
11	0	do.	850-875	60		1.4	2.07	72.7	40.8	Rabbled.
12	0	do.	850-875	40		1.3	1.85	74.5	47.8	Do.
13	0	do.	800-900	60		1.74	Lost	65.0		Reducing atmosphere.
14	0	do.	800-900	60	87.0	1.70	1.70	66.4	51.0	Do.
15	4	do.	975	60		.23	1.11	97.6	69.0	
16	3	3	875	50		1.3	1.75	75.0	50.5	
17	0	6	875	50		3.08	2.00	42.5	43.3	
18	0	6	975	60		.41	1.12	92.2	69.0	
19	0	None	975	60		.11	.70	97.9	80.4	
20	3	3	900	60		.38	.50	92.8	85.0	Ore 10 mesh.
21	3	3	900	60		.35	.22	93.3	93.8	Ore 100 mesh.
22	3	3	900	90		.29	Tr.	94.5	99.9	Ore 100 mesh, 5 per cent silica added.
23	4	4	950	120	85.0	None	.35	100.0	90.5	Half of chlorides added during roast.
24	4	None	850	60	24.5	1.1	.81	83.4	74.5	
25	4	do.	850	80	25.0	1.74	.84	73.5	77.5	
26	4	do.	850	60	89.0	2.36	1.59	62.3	55.4	3 per cent FeS ₂ added.
27	4	do.	850	80	78.0	2.41	1.75	62.0	51.4	Do.
28	3	3	900	120	90.0	.05	.72	99.0	81.0	
29	3	3	900	120	90.0	.78	1.72	81.0	53.0	3 per cent FeS ₂ added. All salt added at first.

TABLE 29. *Data on tests of material from Dry Valley mill dump, Pioche, Nev.*

[Analysis of heading: Pb, 7.12 per cent; Cu, 0.25 per cent; Ag, 10.04 ounces per ton; Au, 0.10 ounce per ton; Fe, 5.36 per cent; CaO, 0.71 per cent; Al_2O_3 , 3.80 per cent; S, 1.08 per cent; insoluble, 78 per cent; size, 90 per cent through 80-mesh. Charge: 100 grains ore, 5 grains water; variable chlorides.]

Test No.	NaCl used.	CaCl ₂ used.	Temperature.	Time.	Calcine.				Percentage of extraction.				Remarks.
					Pb.	Cu.	Ag.	Au.	Pb.	Cu.	Ag.	Au.	
	Per cent.	Per cent.	° C.	Min. utes.	Per cent.	Per cent.	Oz. p. ton.	Oz. p. ton.					
1	6	None	850-900	60	0.58	0.05	5.61	0.05	92.5	81.5	50.5	54.0	Sintered slightly.
2	8	do.	850-900	60	None	Tr.	4.89	.01	100.0	99.9	59.2	67.0	
3	10	do.	850-900	60	.11	.05	7.54	.06	98.6	81.5	33.5	44.9	
4	10	do.	850-900	120	.06	None	4.39	Lost	99.2	100.0	61.1	76.0	Sintered. Half of salt added during roast.
5	10	do.	900	90	None	Tr.	2.13	.02	100.0	99.9	82.1	76.0	
6	8	do.	850-900	60	.07	None	5.59	.05	97.8	100.0	51.6	49.4	
7	8	do.	975	60	.06	None	4.24	.04	99.4	100.0	62.5	63.3	First hour at 800° C.
8	8	do.	975	120	None	None	5.66	.05	100.0	100.0	50.0	44.3	
9	4	do.	900	60	None	.05	2.93	.02	100.0	93.0	73.8	77.1	
10	5	do.	900	180	None	.05	2.14	.02	100.0	93.0	80.9	81.7	Half of salt added during roast. 4 per cent silica added. 10 per cent manganese ore added.
11	5	do.	975	60	.14	None	2.86	.04	98.2	100.0	73.7	56.5	
12	3	do.	900	60	.05	Tr.	4.76	.02	99.3	99.9	59.4	72.5	
13	1	do.	900	120	.30	.04	4.42	.03	96.1	93.0	57.0	70.9	Reducing atmosphere. Reducing atmosphere; 10 per cent manganese ore added.
14	5	do.	900	60	.08	Tr.	1.72	.02	99.0	99.9	84.4	78.0	
15	5	do.	900	135	1.93	Tr.	8.85	.07	64.7	99.9	21.7	24.0	
16	5	do.	900	120	.93	Tr.	2.47	.03	85.9	99.9	75.3	75.0	

TABLE 30.—*Data on tests of material from Horn Silver dump, Frisco, Utah.*

[Analysis of heading: Ag, 6.70 ounces per ton; Au, 0.02 ounce per ton; Pb, 7.4 per cent; Zn, 6.3 per cent; Cu, 0.25 per cent; Fe, 4.4 per cent; insoluble, 53.6 per cent; CaO, 2.2 per cent; Al_2O_3 , 8.5 per cent; S, 6.13 per cent. Salt mixture used, equal parts of NaCl and $CaCl_2$. Theoretical quantity of salt mixture required for Pb, 3.1 per cent; for Zn, 11.4 per cent.]

Test No.	Salt mixture added.	Temperature.	Time.	Weight.	Calcine.			Percentage of extraction.			Remarks.
					Pb.	Zn.	Ag.	Pb.	Zn.	Ag.	
	Per cent.	° C.	Min. utes.	Gm.	Per cent.	Per cent.	Oz. p. ton.				
1	12	850	120	90	0.23	2.36	1.47	97.3	66.6	80.5	Heated from cold state to 850° C. with one-half of salt.
2	12	850	120	91	1.04	3.41	3.89	87.5	50.9	47.7	Heated from cold state to 850° C. with total salt.
3	5	900	120	82	2.79	2.89	4.52	68.7	61.6	44.7	All of salt added to charge, placed directly in hot zone.
4	16	900	120	93	.58	2.36	3.68	93.2	65.0	49.3	Do.
5	16	950	120	93	.76	1.83	3.20	90.5	74.5	58.1	Temperature rose to 1,000° C. Slagged badly.
6	5	950	120	82	.70	1.83	4.00	91.7	76.0	50.7	Temperature rose to 1,000° C. Slagged slightly.
7	12	950	120	90	Tr.	3.2	2.58	99.9	54.0	65.5	One-half of salt added at first. Slagged badly.

TABLE 31.—*Data on tests of material from Bingham Mines Co., Bingham, Utah.*

[Analysis of heading: Ag, 8.96 ounces per ton; Au, 0.01 ounce per ton; Pb, 6.85 per cent; Cu, 0.65 per cent; Zn, 4.53 per cent; Fe, 8.85 per cent; insoluble, 56.3 per cent; CaO, 7.3 per cent; Al₂O₃, 6.2 per cent; S, 1.8 per cent. Salt mixture used consisted of equal parts of NaCl and CaCl₂. Theoretical quantity of salt mixture required for Pb, 3.8 per cent; for Zn, 8.1 per cent.]

Test No.	Salt mixture added	Temperature.	Time.	Calcine.					Percentage of ex- traction.				Remarks.
				Weight.	Pb.	Cu.	Zn.	Ag.	Pb.	Cu.	Zn.	Ag.	
	Per cent.	° F.	Min.	Gm.	Per cent.	Per cent.	Oz. per ton.						
1	10	925-1,000	120	87	Tr.	0.21	2.89	1.71	99.9	71.0	44.4	82.0	Slagged at 1,000° C.
2	10	800-900	120	87	Tr.	.21	2.62	4.5	99.9	71.0	50.8	64.7	One-half of salt added at first, and roasted at 850° C.
3	10	900	120	88	Tr.	.21	1.84	5.20	99.9	71.0	64.0	42.3	All of salt added at first; heated to 900° C.
4	10	900	120	87	Tr.	.16	2.10	4.72	99.9	80.5	60.0	50.7	Roasted at first with no salt, 400° to 800° C.
5	10	900	120	97	0.58	.21	2.10	6.31	93.1	71.0	55.4	25.2	Slagged slightly.
6	10	900	120	86	1.83	.24	3.15	5.51	79.5	70.0	42.0	41.1	
7	10	900-1,000	120	93	1.28	.25	2.48	5.35	82.8	66.0	50.8	38.4	Slagged badly.
8	10	900-1,000	120	87	1.34	.03	3.41	7.10	84.0	95.4	35.5	23.6	Do.

TABLE 32.—*Data on tests of material from Yellow Pine mine, Goodsprings, Nev.*

[Analysis of heading: Ag, 3.9 ounces per ton; Pb, 9.32 per cent; Zn, 29.25 per cent; Fe 2.7 per cent; insoluble, 14.8 per cent; CaO, 6.8 per cent; MgO, 5.5 per cent; S, 0.31 per cent; size, through 10-mesh.]

Test No.	Quantity added to charge.				Time.	Temperature.	Calcine.				Percentage of extraction.				Remarks.
	NaCl.	CaCl ₂ .	FeS ₂ .	Manganese ore.			Weight.	Pb.	Zn.	Ag.	Pb.	Zn.	Ag.		
	Per cent.	Per cent.	Per cent.	Per cent.											
1	5	5	None	None.	120	900	70	6.18	38.9	4.43	53.6	6.9	20.7	Temperature fell to 850° C.	
2	5	5	10	do.	120	900	80	5.8	33.9	3.67	51.8	7.1	24.7	Do.	
3	5	5	None.	10	60	905	75	4.3	37.8	3.48	65.8	2.8	33.0	Temperature rose to 975° C.	
4	5	5	do.	10	120	900	75	6.0	35.5	2.68	57.0	8.6	49.5	Temperature fell to 850° C.	
5	11	11	do.	10	240	900	75	.35	34.1	1.25	97.5	12.2	75.7	Temperature rose to 975° C.	

TABLE 33.—*Data on tests of material from Trapper mill dump, Melrose, Mont.*

[Analysis of heading: Pb, 2.58 per cent; Cu, 0.64 per cent; Zn, 11.32 per cent; Ag, 8.7 ounces per ton, Au, 0.01 ounce per ton; Fe, 7.04 per cent; insoluble, 54 per cent; CaO, 5.5 per cent; Al₂O₃, trace; S, 0.30 per cent; size, through 20-mesh.]

Test No.	Quantity added to charge.				Time.	Temperature.	Calcine.					Percentage of extraction.					Zn leached from calcine.	Remarks.
	NaCl.	CaCl ₂ .	Manganese ore.	Per ct.			Pb.	Cu.	Zn.	Ag.	Au.	Pb.	Cu.	Zn.	Ag.	Au.		
Per ct.	Per ct.	Per ct.	Min. when.	Per ct.	Per ct.	Per ct.	Oz. per ton.	Oz. per ton.						Per ct.				
1	4	4		120	900	Trace	0.17	8.92	0.44	None.	99.9	75.8	33.4	95.7	100	48.7	Still fuming at end of test.	
2	4	4		165	900	None.	.11	9.96	.54	do.	100.0	84.4	25.0	94.7	100	54.0	Size of material, 10 mesh.	
3	5	5		135	900	0.52	.22	8.10	0.35	0.03	82.7	68.7	36.5	37.3	54	53.2	Reducing atmosphere.	
4	5	5	10	120	900	.47	.27	7.30	.48	None.	83.9	60.6	38.8	89.4	8	100	Do.	
5	5	5	10	90	900	.17	.17	9.03	.55	do.	94.2	100.0	24.5	94.1	100			
6	11	6		180	900	None.	.38	4.68	1.2	do.	100.0	52.2	74.1	89.1	100	20.4	Sintered.	

TABLE 34.—Data on tests of material from Hayden mill feed, Clarkdale, Ariz.

[Analysis of heading: Ag, 7.10 ounces per ton; Au, 0.15 ounce per ton; Pb, 1.10 per cent; Cu, 0.45 per cent; Fe, 1.42 per cent; insoluble, 66.2 per cent; CaO, 0.66 per cent; S, 1.2 per cent. size, through 20-mesh.]

Test No.	Quantity added to charge.		Time.	Temperature.	Calcine.					Percentage of extraction.				Remarks.
	NaCl.	CaCl ₂ .			Weight.	Pb.	Cu.	Ag.	Au.	Pb.	Cu.	Ag.	Au.	
	Per cent.	Per cent.	° C.	Min. utes.	Gm.	Per cent.	Per cent.	Oz. per ton.	Oz. per ton.					
1	4	4	900	90	92	Tr.	Tr.	1.37	Tr.	99.9	99.9	82.2	99.9	10 per cent of manganese ore added.
2	4	4	900	105	101	Tr.	Tr.	1.60	0.01	99.9	99.9	77.2	93.3	
3	3	3	900	105	93	Tr.	Tr.	2.57	.02	99.9	99.9	66.5	85.1	

TABLE 35.—Data on tests of material from Sells mine, Alta, Utah.

[Analysis of heading: Ag, 16.35 ounces per ton; Au, 0.04 ounce per ton; Pb, 10.15 per cent; Zn, 6.04 per cent; Fe, 1.98 per cent; insoluble, 6.62 per cent; CaO, 15.75 per cent; MgO, 23.9 per cent; S, trace; size, through 20-mesh.]

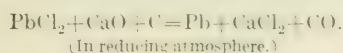
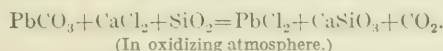
Test No.	Quantity added to charge.			Time.	Temperature.	Calcines.					Percentage of extraction.				Remarks.
	NaCl.	CaCl ₂ .	FeS ₂ .			Weight.	Pb.	Zn.	Ag.	Au.	Pb.	Zn.	Ag.	Au.	
	Per cent.	Per cent.	Per cent.	Min. utes.	° C.	Gm.	Per cent.	Per cent.	Oz. per ton.	Oz. per ton.					
1	5	5		120	900	58	1.33	6.57	5.13	0.015	92.4	36.8	81.8	78.2	10 per cent of manganese ore added.
2	5	5	10	120	900	65	.76	6.04	.36	Tr.	95.2	35.0	98.6	99.9	
3	5	5		150	900	70	Tr.	10.45	8.44	.01	99.9	None.	64.0	82.5	
4	5	5		180	900	64	5.23	6.57	14.30	.015	65.5	30.5	44.2	76.0	

Later tests were made in a small oil-fired mechanical roaster, and in these the volatilized fumes were passed through a single-pipe electrical precipitator and collected. The fume was melted and reduced by heating with additions of calcium oxide and coal dust, a slag of CaCl₂ being obtained.

DISCUSSION OF CONDITIONS OF VOLATILIZATION.

USE OF CALCIUM CHLORIDE.

As calcium chloride may be recovered in the refining process, it would be desirable to make use of that compound as the chloridizing agent. The equations for the reactions are as follows:



From these equations it is seen that as much CaCl₂ is formed as is needed for chloridizing the metal. The amount of sodium chloride necessary for proper volatilization was found to be one and one-half

times the theoretical. Hence, if it be assumed that 75 per cent of the CaCl_2 can be recovered and used, the chloridizing agent used should be a mixture containing equal proportions of NaCl and CaCl_2 . Experiments showed that calcium chloride could replace sodium chloride in the chloridizing action; they also showed that the presence of calcium chloride in approximately the proportion specified was beneficial, doubtless through the formation of calcium silicates which are less fusible at the temperatures used than are the sodium silicates and thus avert the formation of a refractory coating of slag around the particles of ore being chloridized.

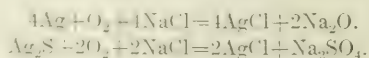
ROASTING TEMPERATURE.

A temperature of 900°C . is necessary for complete volatilization of the gold and the silver, whereas the lead and to a less extent the copper may be driven off at temperatures much lower than 900°C . The chlorides of the metals volatilize at temperatures much below their boiling points. For example, the boiling point of lead chloride has been given as 861 to 954°C . and that of cuprous chloride as 954 to $1,032^\circ \text{C}$., whereas gold and silver chlorides dissociate below these temperatures into the metals and into chlorine. In a chloridizing atmosphere, however, gold and silver chlorides probably exist and are expelled with the other metal chlorides, or oxychlorides. Sodium chloride melts at 805°C . and calcium chloride at 780°C ., so that they are melted and brought into more intimate contact with the ore before the volatilization temperature of 900°C . is reached. Hence they are efficient chlorine carriers.

LENGTH OF ROASTING PERIOD.

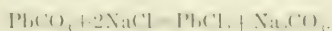
The time necessary for good volatilization depends, of course, on the rate at which the fumes are removed from the surface of the charge and on the area of surface exposed. The former may be increased by forced draft or easy access of air, and the latter by rabbling. If these devices are resorted to, more salt must be used to make up for that lost by increased volatilization.

A strongly oxidizing atmosphere is necessary for good results. Test runs with neutral or reducing atmospheres showed decreased extractions, especially of the gold and the silver. All the tests showed that unless an excess of oxygen was present, the gold and silver volatilizations were too low to warrant the use of the process. That excess oxygen is necessary in volatilizing the gold and the silver is evident from the equations following:



Of course, the Na_2O combines immediately with the silica and other substances in the gangue. As regards lead which exists as PbCO_3 ,

the presence of oxygen seems to be unnecessary, although the lead is so easily reduced that a reducing atmosphere prevents volatilization. The equation for the reaction between lead carbonate and sodium chloride can be written as follows:



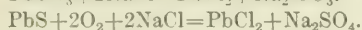
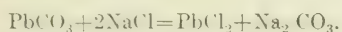
USE OF OXIDIZING AGENTS.

As oxygen is necessary to effect good gold and silver extractions, the presence of an oxidizing agent should hasten volatilization. Tests were run with 5 per cent manganese dioxide in the charge, both in oxidizing and reducing atmospheres; in each test higher extractions resulted.

However, this procedure was tried on a larger scale in a hand-rabbed reverberatory furnace by Mr. Wigton without success. This result may be due to the slow rate of heating of the charge in a reverberatory furnace, the charge being heated so slowly that the available oxygen of the manganese dioxide is gone before the volatilization temperature is reached.

ADDITIONS OF SULPHUR OR SULPHUR COMPOUNDS.

Good extractions were obtained with oxidized ores without the admixture of any sulphur or sulphur compounds. Extractions not so good were obtained with the semioxidized ores treated. Tests of the oxidized ores with sulphur added, in the form of iron pyrites, showed poorer extractions, especially of silver, than tests run under similar conditions without sulphur. These facts lead to the conclusion that the presence of sulphur is not necessary, and is perhaps even harmful. Also more oxygen is required in chloridizing the lead, as is seen by comparing the equations following:



The presence of a large quantity of sulphur or a sulphide would therefore cause lower extractions, as with silver, or necessitate the admission of a larger quantity of air, as with lead.

In Croasdale's tests (U. S. Pat. 741712, Jan. 23, 1900) care was always taken to have enough sulphides (like pyrite) present to permit chloridizing of the valuable metals through the well-known chloridizing-roasting reactions, in which sulphur dioxide reacts on the sodium chloride to liberate hydrochloric acid and chlorine, which attack the valuable minerals. It had been determined that ordinary chloridizing roasting would not be complete unless sulphides were present. Croasdale and his coworkers, and most patentees before and since his time, accepted this fact and always introduced enough sulphide to provide the necessary sulphur for such reactions.

The authors have found that, when the reaction temperature is maintained above the boiling points of any chlorides that might form, the presence of sulphides is not necessary and is a detriment, rather than an advantage. This is possibly a new principle in chloridizing practice, but is based on sound theory. Chemical reactions are often affected by the formation of an insoluble precipitate, a volatile product, or by any set of physical conditions that permits the removal of one of the products of reaction from the reacting mixture. In roasting under the conditions stated, the temperature is held at a point where the metallic chlorides will evaporate; hence it is possible to have sodium chloride or calcium chloride react directly with lead, silver, gold, or copper minerals to form the more volatile metallic chlorides.

Former conceptions of chloridizing have been those necessary for a good chloridizing roast. Chloridizing roasting was carried on at temperatures below the melting points of sodium chloride or calcium chloride, hence the only way to bring the chlorine into intimate contact with the ore particles was by means of the chloridizing atmosphere generated by the reaction of sulphur dioxide and sulphur trioxide, from the oxidation of the sulphides, on the sodium chloride or other solid chlorides present. As regards the process under consideration, the trouble from the presence of sulphur may have been caused by the formation of sulphates of the metals desired, so that more salt would have to be added to react with these sulphates.

CONDITIONS ESSENTIAL FOR GOOD ROASTING.

From the preceding discussion of the conditions of roasting it is seen that four things are of paramount importance—namely: (1) A temperature of 900° C. (1,652° F.); (2) sufficient draft; (3) excess of oxygen; and (4) quick rabbling.

It is not easy to find a furnace that will work economically under these conditions. In some of Croasdale's tests, made in a reverberatory roasting furnace, good extraction was obtained, but the rate of volatilization was slow. A rotary kiln of the type used in cement making might be applicable to the process, as it meets all four of the above requirements, and some such device will probably be the first to be tested on a large scale by some one who may take up the volatilization of ore on a commercial scale. It is possible that at the temperatures desired the atmosphere of a rotary furnace could not be made sufficiently oxidizing, hence the rate of volatilization in such a furnace may also prove too slow. In that event some type of forced-draft grate furnace may be resorted to, and if such a furnace could be constructed that would heat the charge to the desired temperature at reasonable cost and without grate troubles, the success of the process would be assured.

TREATMENT OF FUME.

The advent of the method of electrical precipitation of suspended particles is almost an assurance that the proposed volatilization process will be successful. As previously stated, the principal cause of failure of the Pohlé-Croasdale process was the unsatisfactory fume-collecting device. It is now possible to collect the fume simply and cheaply by the use of an electrical precipitator. In several plants now running, metallic chlorides with other compounds are being precipitated in electrical treaters. The bureau's tests, although conducted on a rather small scale, show that the process is applicable to the chlorides used, almost perfect clearance having been obtained at temperatures in the pipes of 100° to 150° C.

The precipitated fume must be carefully handled in recovering the metals, as it is rather fluffy and easily volatilized. A different treatment than the use of lime and coal for fumes containing copper and zinc might be desirable. However, the main difficulty to be solved is not the treatment of the fume, but the development of a furnace that will insure cheap roasting.

MATERIALS TO WHICH PROCESS MAY BE APPLIED.

The proposed process may be applied to almost any oxidized ore of lead, gold, silver, and copper. Zinc does not respond to such treatment and its action during volatilization is decidedly erratic. Some zinc ores show as high as 75 per cent extraction of zinc and some as low as 10 per cent; with others, to leave most of the zinc in the calcines or volatilize it out was impossible. The composition of the ore had no effect on the applicability of the process, unless the composition was such as to cause slagging at the temperature used. Ores high in silica, iron, lime, magnesia, and alumina all responded readily, giving very good volatilizations. This process may, therefore, be applied to almost any oxidized ore of lead, gold, silver, or copper. It is especially desirable for materials that can not be treated by any of the common methods of gravity concentration, flotation, leaching, or are of too low grade or of undesirable composition to smelt directly.

Tables 28 to 35, inclusive, give the data on the tests made in roasting dishes in a muffle, to determine the range of oxidized and semi-oxidized materials to which this process could apply. It can be seen that some have a high lime content, others are high in iron, others high in silica, etc. The results are largely self-explanatory and conclusions from them have already been given in the foregoing discussion. It was noticed that materials that were finely ground, like the slime from the Horn Silver dump, tend to sinter more easily than when coarsely ground, although they may be far from self-fluxing.

This is probably due to the more intimate mixture of the minerals that are capable of uniting to form slags.

Evidently this method, as it promises successful treatment of a wide range of ores, with less fuel than would be used in smelting and with extractions of the metals that enable it to compete with any other form of hydro- or pyro-metallurgy, and is applicable under the conditions found in arid regions, in relatively inexpensive plants, is sure to be thoroughly tested in the near future.

Since the first writing of this bulletin, chloride volatilization has been further tested on a larger scale in the Salt Lake City laboratory. A miniature cement kiln 7 feet by 1 foot was set up and an electrical precipitation unit to correspond. Better extractions than those obtained in the laboratory were indicated by the lower grade of residue. A great deal of fume escaped through cracks and other openings in the apparatus, but practically all the fume going to the electrical unit was precipitated. A kiln 30 feet by 6 feet in size was constructed during the fall of 1917 by the Chief Consolidated Mining Co. at its mine at Eureka, Utah, under the direction of G. H. Wigton. A similar improvement in extraction was indicated and steps are now being taken to compose a bag house with the electrical precipitator for collecting the fume; also, a larger kiln and precipitator are being built at Fairbank, Ariz., by G. H. Hirt & Co., of El Paso, and will be ready early in 1918.

FLOTATION OF LEAD CARBONATE ORES.^a

As mentioned in foregoing pages, losses of lead carbonate in milling are generally in the slimes, because cerrusite is friable and breaks into small, flat, pancake-shaped particles. Such particles do not settle out, although this mineral has a high specific gravity.

The flotation process is adapted only to the concentration of slimes, hence the thought was immediately suggested that flotation of the slimes containing lead carbonate would correct these losses. However, flotation was known to be successful only with sulphide minerals: hence direct flotation was not immediately adapted to the solution of this problem. Proposals to introduce into a pulp containing oxidized minerals a soluble sulphide, such as hydrogen sulphide, sodium sulphide, or calcium sulphide, in order to form a coating of artificial sulphide of lead on the oxidized particles, have been made in a number of patents. True, the patented processes have usually applied to the flotation of the oxidized minerals of copper, but the wording of most of the patents is broad enough to render application of the process to any other metal liable to claim by the patentee.

^a Experimenters: Glenn L. Allen, E. J. Atkinson, L. D. Yundt, A. J. McChrystal, F. Cameron, and O. C. Ralston.

WORK OF OTHER INVESTIGATORS.

The first patent covering sulphide-filming of oxidized minerals, followed by flotation of the artificial sulphides, was probably that of Schwarz,^a issued in 1905. Schwarz mentions recovery of the artificial sulphide by use of his own method of "bulk oil" flotation with a hydrocarbon, such as paraffin, which is solid at ordinary temperatures, the separation being made in hot solutions and the pulp permitted to cool in order to congeal the paraffin with the entrapped sulphides of lead. He mentions the use of any "soluble sulphide," and states that he generally uses more than the theoretical quantity of sulphur needed to convert the oxide, carbonate, or chloride completely to a sulphide. He claims any method of separating the artificial sulphide which employs concentration by the use of a hydrocarbon, by which one might infer that he can claim the use of froth flotation as at present day developed.

The next mention of artificial sulphidizing and flotation of oxidized minerals is made by Sulman and Pickard^b in British patent 26019, of 1909. In this patent more different methods of sulphidizing the oxidized ore are mentioned and greater familiarity with actual testing of the ores by the proposed processes is shown than in any other sulphidizing patent. Besides oxidized copper ores, oxidized lead ores are mentioned as being adaptable to the process. Hydrogen sulphide and soluble alkaline sulphides are mentioned as possible sulphidizing agents; also a method of sulphidizing by the use of sulphur vapor is described. One variation of the latter method is to heat the powdered ore with FeS_2 in a neutral or reducing atmosphere, distilling one atom of sulphur from the FeS_2 , the freed sulphur combining with the oxidized ore.

Terry^c describes a process for sulphidizing oxidized ores of copper (and other metals) by the use of hydrogen sulphide gas in flowing pulp, with suitable apparatus for recovery of any excess hydrogen sulphide gas after sulphidizing the pulp. A method for the use of sodium sulphide is also described, and of adding copper sulphate to aid the formation of nuclei of copper sulphide around which the formation of sulphide granules and coagulations can take place.

Hovland and Frankforter,^d in their process, use hydrogen sulphide gas on the dry ore to sulphidize the oxidized material previous to flotation. The advantage claimed is the greater rapidity of reaction. From the properties of gases as compared to solutions this claim

^a Schwarz, Alfred, Process of concentrating ores: U. S. patent 807501, Apr. 19, 1905.

^b Sulman, H. L., and Pickard, H. F. K., Improvements in concentration of oxidized ores: British patent 26019, Nov. 10, 1909.

^c Terry, J. T., jr., Process for recovering metalliferous constituents of ores: United States patent 1004760, Sept. 8, 1913.

^d Hovland, H. B., and Frankforter, C. B., Art of treating metalliferous materials: United States patent 1095668, Dec. 12, 1913.

seems justified, because the rate of diffusion of gas in the dry state is thousands of times that of the same gas dissolved in a solution. They state that 10 to 20 minutes' contact of the dry ore with hydrogen sulphide gas is sufficient before flotation. They even claim that the gas might be applied to the ore before mining. The gas, they state, will penetrate the oxidized capping of a body of copper ore, sulphidizing the oxidized forms of copper and disintegrating the mass so that it can be mined easily.

Bacon,^a in 1914, describes a process for using hydrogen sulphide and sulphur dioxide in pulp to aid flotation by means of the colloidal sulphur resulting from the reaction of the two chemicals on each other. The colloidal sulphur acts as a substitute for flotation oil. By introducing the hydrogen sulphide first, oxidized minerals can receive a sulphide coating before the excess of hydrogen sulphide is neutralized by the introduction of sulphur dioxide. This feature is described by Bacon in another patent^b in connection with some tests said to have been made on an oxidized copper ore.

A few months later, Hovland^c took out another patent for the sulphidizing of minerals. A method for the flotation of oxidized copper ore is described. Sulphuric acid is first applied to the pulp to form a solution of copper sulphate. On the addition of calcium sulphide and ferric sulphate to the pulp, copper sulphide forms, which can be recovered by flotation. It is claimed that the reaction is greatly promoted by the ferric sulphate. Without the presence of the ferric sulphate, it is difficult to make the calcium sulphide react.

Bacon^d has also patented a process for the use of hydrogen sulphide under pressure for sulphidizing.

As stated before, practically all of the patents had reference to the oxidized ores of copper and little had been said regarding lead ores, except that various patentees claimed the application to lead carbonates and sulphates. Sulman and Pickard are the only ones that definitely describe the application of these processes to various lead ores. Hence it was decided to test the application of sulphide filming to the oxidized ores of lead, followed by flotation, as a method of ore concentration.

SCOPE OF EXPERIMENTS.

A rather extensive series of tests was outlined in testing various methods of sulphidizing, although a number of the methods were soon found to be impracticable. An outline of the different proposed methods follows:

^a Bacon, R. F., Flotation of minerals: United States patent 1140865, Aug. 14, 1914.

^b Bacon, R. F., Flotation of minerals: United States patent 1140866, Aug. 14, 1914.

^c Hovland, H. B., Method of sulfidizing metals: U. S. patent 1159942, July 16, 1915.

^d Bacon, R. F., Flotation of minerals: U. S. patent 1180816, Aug. 14, 1914.

1. Sulphidizing by treating the dry crushed ore with hydrogen sulphide gas.

2. Sulphidizing by leaching the crushed ore in a solution of hydrogen sulphide or by bubbling hydrogen sulphide through the suspended pulp.

3. Sulphidizing with solutions of the various sulphides of sodium.

4. Sulphidizing with solutions of the various sulphides of calcium.

5. Sulphidizing by passing sulphur vapor through the dry crushed ore.

6. Sulphidizing by flotation with a flotation oil containing loosely combined sulphur in the oil molecule. Such oils may be called "sulphureted" oils.

7. Sulphidizing with colloidal sulphur solutions.

For each of these methods many tests would be necessary to determine the best length of time to apply the sulphidizing agent, whether the mineral formed needed to be floated in neutral, alkaline, or acid solutions, whether certain flotation oils would be better than others in flotation of the filmed sulphides, and other problems. The complete testing of all these variables would be an immense undertaking, and therefore many "short cuts" to results that were worth while had to be used. This called for a great deal of the "cut and try" type of research work, and the results are hence difficult of tabulation. It is almost necessary that a flotation test man be an opportunist and when he sees a test yielding poor results to change the flotation or other conditions immediately, in order to save a test that might otherwise prove to be only the waste of an hour's time.

SULPHIDIZING WITH DRY HYDROGEN SULPHIDE.

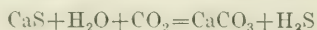
Hydrogen sulphide, as a metallurgical reagent, was used as a precipitant in the old chlorination processes of extracting gold from its ores, but since the displacement of chlorination by cyanidation it has fallen into disuse. The development of the hydrometallurgy of copper gave some promise of reviving its use, but both electrolytic and scrap-iron cementation methods of precipitation of copper permit the direct production of metallic copper, so hydrogen sulphide was again discarded.

FORMER ATTEMPTS TO USE HYDROGEN SULPHIDE.

In spite of the fact that the use of hydrogen sulphide has been repeatedly introduced and discarded by the metallurgical profession, this reagent is still of good repute among chemical engineers, because it can be produced cheaply. The chief reason for not using hydrogen sulphide has not been the cost of production, but rather the fact that better metallurgical methods made its use unnecessary.

The best-known method of making hydrogen sulphide gas is to treat ferrous sulphide (iron matte) with a dilute solution of sulphuric acid. Iron matte can be made in almost any smelter, by distilling half the sulphur from pyrite, at a cost not to exceed \$5 per ton of matte. Sulphuric acid is available in most mining districts at \$5 to \$25 per ton, so that the ultimate cost of hydrogen sulphide per ton of gas, everything considered, will be \$30 to \$60.

Another old method of making hydrogen sulphide is by the Claus-Chance process, in which calcium sulphide is treated with carbon dioxide gas in one form or another, according to the reaction:



Still another method of making hydrogen sulphide is by passing sulphur vapor and hydrogen or hydrocarbon gases through a heated vessel in the presence of the proper catalytic agents, such as charcoal. About 450° C. is said to be the most favorable temperature.

During 1914 and 1915 a number of attempts were made to use hydrogen sulphide in some of the concentration mills of the Southwest where sulphide ores of copper carrying some oxidized minerals were being concentrated. The gas was usually introduced directly into the flotation machines and the atmosphere of the mills, as a result, became unbearable. Hydrogen sulphide gas acts like a narcotic poison and the men working in it finally suffered from nervous demoralization. Any use of such a chemical in a mill must be made in machines that will not permit it to escape into the atmosphere of the mill. Successful machinery for this use is described by Callow.^a

RESULTS OF TESTS AND DISCUSSION OF RESULTS.

The results of applying dry hydrogen sulphide gas to dry crushed ore, for three different Utah ores, are given in tables 36 and 38.

The tests in Table 36, on material from the May Day mine, were made to investigate a number of points. Tests 1 to 5 and tests 6 to 10 were made respectively by dry sulphidizing followed by flotation in neutral and acid solutions, with the idea of determining what length of treatment of the ore with the hydrogen sulphide gas was necessary.

The results of the first five tests show that treatment of the ore with hydrogen sulphide gas in the dry state, followed by flotation in neutral solutions, did not give satisfactory extractions. The best results in these tests were with prolonged treatment with hydrogen sulphide. Considerable heat was generated by the reaction and the ore was quickly blackened by the action of the gas. A well sulphidized sample of ore could be poured through the air after sulphidizing with the result that it as quickly reoxidized. The iron analyses show that some of the iron was also sulphidized and accompanied the lead.

^a Callow, J. M., Notes on flotation, 1916; Bull. Am. Inst. Min. Eng., 1917, pp. 245-275.

TABLE 37.—*Results of sulphidizing material from Chic Consolidated mine with dry hydrogen sulphide.*

[Assay of heading: Pb, 3.12 per cent; Ag, 6.88 ounces per ton; Zn, 2.65 per cent; Fe, 6.35 per cent; Insoluble, 68.5 per cent.]

Test No.	Quant-ity of ore used	Kind.	Quan-tity, pounds per ton.	Acid used, pounds per ton.	Time of sul-phid-izing.	Weight of froth.	Analysis of concentrate.										Remarks.
							Pb.	Ag.		Zn.		Fe.	Insoluble.				
								Per-cent.	Per-cent. of ex-trac-tion.	Ounces per ton.	Per-cent. of ex-trac-tion.		Per-cent.	Per-cent. of ex-trac-tion.	Per-cent.	Per-cent. of ex-trac-tion.	
1	500	Crude cedar.....	3.2	73.6	2	24.0	11.6	17.8	31.66	22.2	4.9	9.0	15.7	31.0	2.2	Second fraction, additional acid used. First fraction. Second fraction. Third fraction, additional acid used.	
2	500	do.....	3.2	184	2	37.0	11.2	26.5	31.64	34.1	4.4	12.4	8.7	10.1	3.8		
3	500	do.....	3.2	405	2	62.0	9.0	35.8	30.88	55.7	3.9	18.4	10.2	19.9	5.9		
4	500	Pine.....	2	368	2	116.0	8.9	65.2	22.1	74.6	3.0	26.5	8.3	30.3	50.4		
5	500	do.....	4	368	2	30.5	7.5	24.3	45.4	66.6	2.4	9.2	9.0	14.3	5.3		
6	500	do.....	6	368	2	65.5	11.1	46.6	32.2	59.8	2.8	14.0	7.4	15.3	46.2		
7	500	do.....	2.0	368	2	55.5	4.0	14.2	34.1	55.0	3.5	14.8	9.7	17.0	4.0		
8	500	Crude cedar.....	3.2	184	15	22.5	7.9	11.4	45.1	29.5	4.1	7.0	11.7	8.3	2.2		
9	500	do.....	3.2	184	30	20.5	11.5	15.1	51.9	31.0	3.8	5.9	11.7	7.6	1.6		
10	500	do.....	3.2	184	2	37.0	11.2	26.5	31.6	34.1	4.4	12.4	8.7	10.1	3.8		
11	500	do.....	3.2	184	8	33.0	11.8	25.0	35.6	34.2	5.2	13.1	14.0	14.5	26.6		
12	500	do.....	3.2	184	16	33.0	11.8	25.0	27.8	26.6	4.4	11.1	9.8	10.2	2.6		
13	500	do.....	3.2	184	16	46.5	11.5	34.3	31.2	42.1	4.2	14.9	15.4	22.6	30.4		
14	500	Pine.....	8	294	4	8.5	6.8	3.7	20.4	5.0	4.2	2.7	13.0	3.5	26.6		
15	500	do.....	8	73		30.5	5.0	9.8	19.3	17.1	3.2	12.8	11.6	11.1	26.8		
15	500	do.....	8	73		25.0	8.2	13.1	25.2	18.4	3.4	6.5	7.4	34.0	2.5		

Second fraction, additional acid used.

First fraction.
Second fraction.
Third fraction, additional acid used.

TABLE 38.—*Results of sulphidizing Daly-Judge carbonate ore with dry hydrogen sulphide.*

[Assay of heading: Pb, 16.54 per cent; Ag, 19.90 ounces per ton; Zn, 5.37 per cent; Cu, 0.02 per cent; Fe, 5.3 per cent.]

Oil used.			Acid used, pounds per ton.	Period of sul- phidizing.	Weight of froth.	Analysis of concentrate.										Remarks.			
Quantity of ore used.	Kind.	Quantity, pounds per ton of ore.				Pb.		Ag.		Zn.		Cu.		Fe.			Insoluble.		
						Per- cent.	Ounces per ex- trac- tion.	Per- cent- age of ex- trac- tion.	Per- cent.	Per- cent- age of ex- trac- tion.	Per- cent.	Per- cent- age of ex- trac- tion.	Per- cent.	Per- cent- age of ex- trac- tion.	Per- cent.		Per- cent- age of ex- trac- tion.	Per- cent- age of ex- trac- tion.	
Grams.				Hours.	Grams.														
1	500	Idaho pine	0.8	184	None.	68.5	33.0	19.6	45.1	29.9	10.5	27.2	1.6	35.6	10.7	26.2	21.4	5.4	
2	500	do.	.8	184	2	123.5	30.1	46.0	52.7	62.7	7.7	35.9	1.8	72.8	10.8	47.6	14.8	6.7	
3	500	do.	.8	184	2	57.0	25.5	18.1	37.8	20.8	6.3	13.6	1.0	18.8	8.2	16.7	25.4	5.3	
4	500	do.	.8	184	4	49.5	10.5	6.5	28.8	13.8	5.4	10.1	.9	14.7	6.3	28.5	40.8	7.4	
5	500	do.	.8	184	8	98.5	21.2	25.9	36.0	34.2	5.8	21.5	1.7	40.2	8.1	28.5	25.6	9.2	
6	500	do.	.8	184	8	70.0	27.7	24.1	36.2	24.6	4.5	11.9	1.7	40.2	7.3	18.3	23.4	6.0	
7	500	do.	.8	37	2	66.0	26.0	21.3	48.3	30.8	5.7	14.2	.9	19.6	10.5	24.8	21.4	5.2	
8	500	do.	.8	110	2	67.5	26.7	22.4	49.4	32.2	4.4	11.2	1.0	21.3	9.2	22.2	25.0	6.2	
9	500	do.	.8	187	2	49.5	10.5	6.5	28.8	13.8	5.4	10.1	.9	14.7	6.3	28.5	40.8	7.4	
10	500	do.	.8	368	2	190.0	33.6	83.2	41.5	80.0	5.8	43.6	1.1	72.3	7.8	55.5	22.0	16.0	
11	500	do.	.8	None.	2	83.0	27.4	28.3	40.7	32.7	6.0	18.8	.9	24.7	13.6	40.3	19.0	5.8	Used 7 c.c. ammonia water.
12	500	Eucalyptus.	124.0	368	None.	52.0	30.3	19.6	53.7	27.1	14.5	28.5	.9	15.1	13.0	24.1	9.8	1.9	Tails of test 11, retreated.
13	500	Coal creosote.	26.0	None.	2	49.5	16.9	10.4	61.0	29.2	2.5	4.7	Tr.	6	1.9	3.4	8.2	1.5	Melting from test 12.
14	500	do.	.2	None.	2	22.0	28.7	10.9	23.8	7.0	11.1	10.0	2.3	23.8	3.2	2.5	16.0	1.3	Heating from test 12, ex- treated.
15	500	Coal creosote.	.4	368	1	30.5	28.7	10.9	23.8	7.0	2.2	2.5	Tr.	6	5.2	5.7	38.2	4.3	Tailing of test 14.
16	500	do.	.4	368	1	287.5	39.0	14.2	30.8	8.5	Tr.	Tr.	Tr.	6	2.5	25.6	87.6	86.0	
17	500	Coal creosote.	10.0	184	2	107.0	26.9	35.7	49.5	51.7	8.9	36.0	1.4	49.8	11.2	42.8	12.2	5.2	Ground to 60 mesh.
18	500	do.	1.4	184	2	113.0	28.2	50.1	41.7	57.6	6.9	37.2	.7	31.4	9.4	48.0	22.6	11.8	Ground very fine in ball mill.
19	500	Idaho pine	.8	184	4	179.0	26.1	58.2	34.7	60.2	6.1	41.2	1.4	80.5	8.9	54.9	28.0	18.4	Do.

First fraction.

Second fraction.

Used 7 c.c. ammonia water.

Tails of test 11, re-treated.
Melting from test 12.
Fusing from test 12, re-treated.

Tailing of test 14.

Ground to 60 mesh.
Ground very fine in ball mill.

Do.

The results of tests 6 to 10 show plainly that treatment of the dry ore with the dry gas, followed by flotation in an acidified solution, gives much cleaner concentrates and better extractions than flotation in a neutral pulp. Less iron was found in the concentrates, and it was noticed that on addition of the acid solution to the pulp in the flotation machine hydrogen sulphide gas was always evolved in considerable quantity. This result is not surprising, as iron sulphide will react with dilute acid solutions, whereas lead sulphide is more stable. The presence of the hydrogen sulphide in the pulp did not seem to affect the flotation of the artificial sulphide, although flotation men often say that the flotation of the natural sulphides is deleteriously affected by hydrogen sulphide. The results of tests 11 and 12, with a different oil, were about the same as those of the previous tests. It can be seen that 4 to 8 hours' treatment of the dry ore with hydrogen sulphide gas, followed by flotation in an acid solution, are the necessary conditions for good extractions on this ore and that neutral flotation is not satisfactory.

Tests 14 to 21 were with different flotation oils and no acid, to determine whether any of these oils would give clean concentrates and good extractions without the addition of acid. It can be seen that none were satisfactory. The pine oil and the cedar oil were the best ones.

The results of test 23, compared with those of test 22, show the effect of extremely fine grinding as compared with ordinary grinding for flotation. Most of these tests were made on material ground to pass a 0.25 mm. (60-mesh) sieve, whereas test 23 was made with material which had been ground in an Abbé pebble mill to an impalpable powder before sulphidizing. The extraction of lead was much better with the finely-ground material, although still far from satisfactory. When acid was added, in tests 6 to 10, it is probable that the large amount of acid used up by the ore helped to disintegrate the ore in the same way as fine grinding, allowing more particles of lead carbonate to be acted on.

The last three tests of the series show the effects of increasing the amount of acid used during flotation, after 2 hours of sulphidizing. The higher extractions obtained by using the larger amounts of acid is plainly evident.

In similar tests (Table 37, p. 90) of material from the Chief Consolidated mine of the Tintic district, practically the same results were obtained. The first three tests showed that increasing the proportion of acid used in the flotation solution increased the extractions of lead and of silver. In the second series, tests 4 to 7, the effect of using different proportions of the same flotation oil was tested. Test 7 shows plainly the effects of "over-oiling." The third set, tests 8 to 12, to

find the effect of time of treatment with hydrogen sulphide showed that this ore required about two hours' treatment. The last three tests were made to determine whether the froth coming off at the beginning of the test was any richer than that coming off later in the test. The results show that it was the further addition of acid that affected the tenor of the froth concentrate, and not the time at which it was sampled.

The silver extractions from the Chief Consolidated ore by dry sulphidizing and flotation were always higher than the corresponding lead extractions, whereas with the May Day ore the reverse was true.

The results of 18 tests of the carbonate shipping ore from the Daly Judgemine of Park City, Utah, are shown in Table 38, page 91. This ore resisted all efforts at concentration by this method until an excessive amount of sulphuric acid, 368 pounds per ton of ore (see test 9), was used in the flotation.

SUMMARY OF CONCLUSIONS FROM RESULTS OF DRY SULPHIDIZING.

The conclusions from the tests of dry sulphidizing of lead carbonate ores with hydrogen sulphide gas, followed by flotation of the artificial sulphides, may be summarized as follows:

1. The ore must be treated with the hydrogen sulphide gas 2 to 8 hours, although the reaction takes place quickly and with the development of considerable heat. A tumbling barrel is best suited for applying the gas.

2. A considerable excess of hydrogen sulphide is consumed. The gas seems to penetrate to the centers of the ore particles, and some of the oxidized iron minerals are likewise sulphidized.

3. Silver minerals associated with the lead carbonate, or separate from the lead carbonate, usually float when the ore is treated in the flotation machine.

4. Satisfactory extractions and satisfactory grades of flotation concentrate are obtained only when a considerable excess of sulphuric acid is applied to the sulphidized ore while in the flotation machine. The greater proportion of the artificial iron sulphide formed during sulphidizing is acted on by the acid, with generation of hydrogen sulphide gas in the flotation pulp. This gas does not seem to affect deleteriously the flotation of the artificial sulphides.

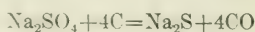
5. In view of the fact that so much hydrogen sulphide and sulphuric acid are necessary in the treatment of the ores tested by this method the writers do not believe that this phase of sulphidizing and flotation would be a commercial success with average ore.

SODIUM SULPHIDE FOR SULPHIDIZING.

SULPHIDE COMPOUNDS AVAILABLE FOR COMMERCIAL USE.

There are three sulphide compounds of sodium that give promise of commercial application in sulphidizing ores—the normal sulphide, Na_2S ; the polysulphides, Na_2S_4 and Na_2S_5 ; and the sulph-hydrate, NaSH .

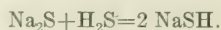
The ordinary commercial method of preparing normal sodium sulphide is from the sulphate of sodium by reduction with carbon, the reaction being as follows:



The freshly reduced chemical is infusible (melting point, approximately $2,000^\circ \text{C}.$), and hence is a porous cinder when drawn from the furnace. This product is subject to spontaneous combustion in air, and is usually wet with enough water to form one of the hydrates of sodium sulphide. Two commercial grades can be bought, the "60 per cent" and the "30 per cent," depending upon the proportion of water of crystallization. Probably the largest use of sodium sulphide at present time is in removing hair from hides in tanneries. It can usually be bought for \$40 to \$30 or less per ton at the factories, but owing to the abnormal conditions since 1914, its price has risen to \$80 to \$100 a ton.

The polysulphides of sodium can be made by boiling powdered sulphur in solutions of caustic soda. Most of the sulphur combines to form Na_2S_4 and Na_2S_5 , although some thiosulphates, thionates, etc., are formed. This type of solution seems to be more sluggish than the normal sulphide of sodium and probably only one of the sulphur atoms in the molecule functions as a sulphide ion, the others being released as colloidal sulphur.

The sulph-hydrate of sodium, NaSH , is formed from the normal sulphide in solution by hydrolysis and seems to be the most active of these three compounds in the sulphidizing of lead carbonate ores. It can be prepared from the normal sulphide by treating the sulphide with hydrogen sulphide gas, the reaction being:



This would make it more expensive than the normal sulphide when prepared in the pure state. The hydrolysis of the normal sulphide gives sodium hydrate as well as the sulph-hydrate, as follows:



The presence of this sodium hydroxide may explain why pure sulph-hydrate of sodium will seemingly be more efficient in its action in sulphidizing a lead carbonate ore, although the explanation is not

immediately clear. Results of tests indicate that the advantage of using sulph-hydrate, as compared with the normal sulphide, was not enough to make the separate preparation of the sulph-hydrate worth while.

ACTION OF SULPHIDES OF SODIUM ON THE ORE.

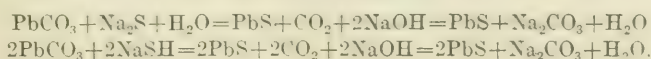
When a solution of any of these compounds is applied to ground ore, the ore slowly turns black. Slightly better results in the flotation were obtained when the ore was allowed to stand long enough to become thoroughly blackened by the sodium sulphide. The artificial lead sulphide, on standing, finally assumed a crystalline appearance, resembling galena. It was noticed during the tests that sulphidized charges which were set aside for a number of days before flotation often gave better results than the ones that were treated immediately after sulphidizing. Whether the artificial lead sulphide actually recrystallizes on standing is not known, but the results seem to indicate that it does.

Some of the most successful tests in the sulphidizing of lead carbonate ores followed by flotation of the artificial sulphide were with the normal ('60 per cent') sodium sulphide of commerce.

DESCRIPTIONS OF TESTS AND RESULTS.

The results of a few tests of the most amenable ore tried are contained in Table 39. This ore was from the Shattuck mine, Bisbee, Ariz., where a large cave-in had opened up a considerable amount of it.

The table shows that 5 pounds of sodium sulphide per ton of ore was not enough for satisfactory extraction of the lead, but when 10 pounds was used per ton, applied in a $\frac{1}{2}$ per cent solution for two hours of contact, 93.2 per cent of the lead in the ore was recovered as concentrate containing 37.3 per cent lead. The use of larger amounts of sodium sulphide merely tended to increase the grade of the concentrate, the extraction of the lead and the silver not being greatly increased. The advisability of using more than 10 pounds of sodium sulphide per ton would depend upon local conditions and ore contracts. A long freight haul of the concentrates would probably make it advisable to use more sodium sulphide and to obtain higher-grade concentrates. In each test the ore seemed to use all the sulphur in the sodium sulphide, but the solution was often alkaline after treatment, so that the excess of alkaline solution would have to be decanted before flotation and the thickened pulp thinned with fresh water. The reaction with the ore is probably according to one of the equations following:



The ore seems especially adapted to flotation, as the silver and gold in the ore accompany the lead and permit high extractions and good grades of concentrate. In fact, few present ore-dressing operations show an extraction of more than 90 per cent of the metal sought, as was obtained in the flotation tests of this ore.

Table 40 shows the results of sodium sulphide in sulphidizing two other lead-bearing materials that contained very little silver. One was material from the Scranton mine in the North Tintic district, Utah, and the other was tailings from the Wilbert dump near Arco, Idaho. From the Scranton ore more than 90 per cent of the lead was extracted in the form of a concentrate carrying 50 to 65 per cent lead. A mixture of coal-tar creosote and wood turpentine seemed to be a satisfactory flotation oil. Twelve pounds of sodium sulphide per ton of ore, applied in a weak solution for one-half hour, seemed to be satisfactory. The heading sample contained 9 per cent lead, or 180 pounds, which would require 68 pounds of sodium sulphide to convert the lead completely to sulphide, as only 12 pounds was consumed, all of the lead floated could not have existed as lead sulphide. Probably only the surfaces of the lead carbonate particles had been converted to sulphide of lead. This has been termed "sulphide filming." The Shattuck ore must have had an even thinner film of sulphide than the Scranton ore. More recent tests made by G. L. Allen, while in the employ of the Shattuck-Arizona Copper Co., have shown good extractions with as low as 2 pounds of sodium sulphide per ton of ore.

The Wilbert tailing was more difficult to treat successfully, as the extractions were usually low and the grade of concentrate rarely above 30 per cent lead. This tailing is from a concentrating mill and carries only 5.5 per cent lead.

Coal-tar creosote combined with wood turpentine, pine oil, or cedar oil made a suitable frothing agent for these materials.

Results of further tests with sulphide filming by means of sodium sulphide are shown in Tables 41 and 42. The material from the Bullion Beck slime dump illustrates one type of material that often resists all efforts to extract the lead efficiently by sulphide filming. Seemingly some compound or compounds in the material used up the sodium sulphide before it could blacken the particles of lead mineral. Usually an excess of the reagent would permit better extractions, but the consumption was too large for commercial work. Results of tests of some other materials of this kind are recorded in Table 43 (p. 104). Nearly all of these materials contained noteworthy proportions of colloidal alumina compounds or of colloidal limonite. The best results in sulphide filming with sodium sulphide were obtained with those materials that did not have such a large colloidal content.

TABLE 39.—*Results of sulphidizing ore from Shattuck mine with sodium sulphide.*

[Analysis of heading: Pb, 15.42 per cent; Ag, 12.88 ounces per ton; Au, 0.05 ounces per ton.]

Test No.	Quantity of ore used.	Na ₂ S used.		Time of sulphidizing.	Oil used, pounds per ton of ore treated.	Concentrates.							
		Quantity, pounds per ton of ore.	Strength of solution, per cent. Na ₂ S.			Lead.		Silver.		Gold.		Iron content.	Insoluble content.
						In concentrate.	Extraction, percentage of total content in ore.	In concentrate.	Extraction, percentage of total content in ore.	In concentrate.	Extraction, percentage of total content in ore.		
						Percent.		Ounces per ton.		Ounces per ton.		Per cent.	Per cent.
1	500	5	0.25	11. m.	Refined turpentine, 0.4; coal-tar creosote, 1.2; and eucalyptus, 0.2 pound.	34.5	36.9	43.10	51.8	0.11		3.1	44.6
2	500	10	.50	2	do.	37.3	63.2	32.70	88.9	.10		3.3	43.8
3	500	15	.75	2	do.	39.1	84.8	32.96	86.5	.10		3.6	44.0
4	500	20	1.00	2 40	Refined turpentine, 0.3; hardwood creosote, 0.9 pound.	50.75	97.75	39.92	88.8	.14		3.50	28.6
5	500	40	2.00	1 30	Coal-tar creosote, 2.14; turpentine, 0.24; and pine oil, 0.05 pound.	70.1	96.25	58.34	93.5	.22		1.27	6.4

TABLE 40.—*Results of sulphidizing ore from Scranton mine and material from Wilbert mill dump.*
ORE FROM SCRANTON MINE.

Test No.	Quantity of material taken for test.	Na ₂ S used.		Time.	Oil used, pounds per ton of material.	Concentrates.	
		Quantity, pounds per ton.	Strength of solution, percentage of Na ₂ S.			Lead content.	Extraction of lead.
				Hours.		Percent.	Percent.
1	500	12	0.6	4	Refined turpentine, 0.2, and coal-tar creosote, 1 pound.	65.5	89.7
2	500	20	1	1½	"Crescedar," ^a 0.72 pound.	29.5	90.4
3	500	40	2	2	Coal-tar creosote, 1.2, and refined turpentine, 0.2 pound.	53.7	91.2
4	500	40	2	1	"Crescedar," refined turpentine, coal-tar creosote, and rosin, 2 pounds.	66.5	84.3
MATERIAL FROM WILBERT MILL DUMP.							
5	500	5	0.25	18	Crude coal tar, 1.5, and pine oil, 0.6 pound.	16.5	52.4
6	500	10	0.5	18	Pine oil, 0.6 pound.	27.3	31.3
7	500	10	0.5	3½	Refined turpentine, 0.2, and crude coal-tar creosote, 1 pound.	33.15	79.4
8	500	20	1	2	Turpentine and S ₂ Cl ₂ , 0.8, and crude coal-tar creosote, 0.8 pounds.	27.2	57.2

^aConsists of 3 parts of coal creosote to 1 part of cedar oil.

TABLE 41.—Results of sulphidizing material from Bullion Beck slime dump with sodium sulphide.

[Assay of heading: Pb, 5.5 per cent; Ag, 7.2 ounces per ton; Au, 0.02 ounce per ton.]

Test No.	Quantity of material taken.	Na ₂ S used.		Time.	Kind.	Oil used.	Concentrates.						
		Quantity, pounds per ton of material treated.	Strength of solution, percentage of Na ₂ S.				Quantity, pounds per ton of material.	Lead.		Silver.		Gold.	
								In concentrates.	Extraction, percentage of total content in ore.	In concentrates.	Extraction, percentage of total content in ore.	In concentrates.	Extraction, percentage of total content in ore.
	Grams.			Hours.			Percent.		Ounces per ton.		Ounces per ton.		Ounces per ton.
1	500	20	1	0.5	Turpentine		34.2	71.7	21.8	36.4			
2	500	20	1	1	do.		35.0	44.5	28.1	27.3			
3	500	20	1	1.5	do.		30.8	41.4	35.9	36.9			
4	500	20	1	2	do.		36.4	46.3	29.5	38.7			
5	500	20	1	3	do.		34.6	57.7	28.2	23.5			
6	500	20	1	10.5	do.		33.0	59.4	26.2	36.0			
7	500	20	1	17	do.		19.4	62.4	18.7	46.0			
8	500	40	2	17	Pinewood oil saturated with sulphur		27.0	68.7	27.8	54.2			
9	500	100	5	88	do		20.0	63.0	19.3	46.6	0.67	6.4	
10	500	20	1	24	Turpentine		13.1	79.0	13.1	54.5	.05	78.0	
11	500	20	1	17	do		49.4	62.4	18.7	46.9			

TABLE 42.—Average results of sulphidizing miscellaneous materials.

Kind.	Material tested.			Analyses of concentrates.			Extraction.			N ₂ S used, per ton of material treated.	Time of treat- ment.
	Lead content.	Silver content.	Gold content.	Pb.	Ag.	Au.	Pb.	Ag.	Au.		
										Per cent.	Ounces per ton.
Ore from Chief Consolidated mine, Utah.....	2.8	8.0		8.7	31.9		68	45		10	3.5
Slime from Eureka Hill dump, Utah.....	2.0	4.7		14.0	16.9	0.07	40	20	30	20	28.0
Ore from American Flag mine, Utah.....	4.2	18.12	0.02	27.1	216.0	1.33	43	48	40	12	3.0
Tailing from Bullionville dump, Nevada.....	9.87	11.12	.19	35.9	14.8	.16	63	46	40	6	2.0
Tailing from Dry Valley dump, Nevada.....	7.12	10.04	.10	57.8	23.2	.10	76	27	31	20	5.0
Material from Yellow Pine mine, Nevada.....	15.04	11.92		48.3	29.64		89.2	51.7		20	1 1/2
Tailing from Ontario dump, Utah.....	4.92	9.44	.04	32.5	35.78	.22	62	45	20	20	2
Daly West dump, Utah.....	5.25	21.8	.02	9.27	58.70	.02	15	44		20	2

DISCUSSION OF RESULTS.

The tables show that the extractions of silver, when present, were usually lower than the extractions of the lead. This result was unexpected, as the writers had been led to believe that the silver in such materials was intimately associated with the lead. Grinding the sample to the fineness necessary for flotation (passing 60 mesh) evidently freed the silver to a large extent, as far as these results are concerned.

The crushed ore could probably be treated with sodium sulphide solution in Dorr thickeners by adding solution to the inflow. The overflow of clear solution, containing some sodium sulphide, could be brought to the proper strength by adding strong sodium sulphide solution from a drip, and returned to join the inflow. If the solution should become too alkaline, it would have to be either thrown away or neutralized and oxidized. Tests to determine the maximum alkalinity permissible showed that solutions containing proportions ranging up to 0.3 per cent of sodium hydroxide could be successfully used in flotation of many ores, but that with more than 0.3 per cent the froth was often too tough and leathery and the flotation was not selective. When a longer time of contact with the sodium sulphide solution than that necessary to pass through the Dorr thickener was required, some form of agitator that does not utilize a jet of air would be necessary. Tests of agitating a pulp with air while sulphidizing showed serious oxidation of the sodium sulphide and of the artificial lead sulphide, so that practically no blackening of the particles took place. A series of Dorr classifiers or a tube mill containing no grinding balls are machines that could meet the requirements of nonaeration combined with agitation of the pulp.

The interfering elements that prevent successful sulphide filming of lead carbonate ores were gradually isolated during the course of this investigation. In a number of instances materials were tested that would give no extraction of lead by sulphide filming and flotation. From the flotation tailing a streak of white lead carbonate could be separated on a gravity concentration table. This carbonate had seemingly not been sulphidized, some constituent in the ore combining with the sodium sulphide before it had time to act on the lead. A number of the minerals that caused trouble were identified. In the ore from the Chief Consolidated mine of Eureka, Utah, it was possible to identify a large amount of basic sulphates of iron, such as utahite and jarosite. These combine vigorously with sodium sulphide and become black, but are nonfloating. Manganese dioxide minerals also combine vigorously with sodium sulphide, oxidizing it to thionates and thiosulphate, and hence cause trouble. Enough sodium sulphide to satisfy these compounds must be added before

the lead minerals can be sulphidized. In the Michigan-Utah mine in Alta, Utah, is a lead carbonate ore containing both manganese dioxide and basic sulphates of iron. Attempts to remove these compounds have thus far been unsuccessful, because the basic sulphate of iron is insoluble in water, as is also the manganese dioxide. The use of acids to remove them is not practicable, owing to the high percentage of lime and other acid-consuming elements usually present in these ores. Sulphur dioxide will "bleach" the manganese dioxide, but it forms di-thionates, which are deleterious to flotation. The presence of these minerals, which are fairly common, owing to the fact that they have their origin in oxidation of sulphide ores, probably explains the difficulty of floating certain carbonate ores of lead. Where an ore is not refractory to sulphidizing and flotation, this method is a useful one.

CONCLUSIONS AS TO RESULTS OF TESTS WITH SODIUM SULPHIDE.

The conclusions reached from the tests with sodium sulphide as a sulphidizer previous to flotation are as follows:

1. Small quantities of sodium sulphide, varying from 2 to 40 pounds per ton of ore, will convert the lead carbonate of ordinary low-grade oxidized lead ores to a condition amenable to flotation.

2. The amount of sodium sulphide consumed is so small that evidently only a lead sulphide film is formed over the surfaces of the particles of lead carbonate.

3. The time of contact of the sodium sulphide with the ore should be varied according to the strength of the solution used and the properties of the ore. With a 1 per cent solution usually about two hours is required, although with some ores a much shorter period of contact suffices. Much better results are obtained when the sulphidized ore stands for a considerable length of time before flotation.

4. Air agitation during sulphidizing is to be avoided on account of oxidation of the sodium sulphide and of the lead sulphide.

5. Ores containing large amounts of colloidal alumina or iron, or basic sulphates of iron or alumina, or manganese dioxide minerals, are difficult to sulphidize and more difficult to treat by flotation. So far they have not been treated successfully by this method. Most oxidized ores contain one or the other of these deleterious constituents.

6. When silver minerals are present in the ore, the extraction of silver is usually 5 to 15 per cent lower than the extraction of lead.

7. Commercial sodium sulphide can be bought at a reasonable price, and the quantity required to sulphidize a ton of ore is small,

hence this phase of sulphidizing and flotation is distinctly promising from a commercial standpoint.

8. The polysulphides of sodium and the sulphydrate are acceptable substitutes for the normal sulphide, although they are usually not for sale on the market. The sulphydrate seems to be the most active sulphidizing agent of all the sulphur compounds of sodium tested.

CALCIUM POLYSULPHIDE FOR SULPHIDIZING.

COMPOUNDS AVAILABLE FOR COMMERCIAL USE.

The sulphidizing compounds available for commercial use that have calcium as a base are the normal sulphide, CaS , the polysulphides, CaS_5 and CaS_6 , and the sulphydrate, $\text{Ca}(\text{SH})_2$. They are prepared in exactly the same way as the sodium compounds, and, similarly, the sulphydrate has been found to be the most active. The normal sulphide of calcium is a by-product in the Le Blanc process of soda manufacture, but unfortunately is only slightly soluble. It can also be produced by the reduction of gypsum with carbon. The polysulphides of calcium can be prepared from quicklime and powdered sulphur and have the further advantage of being extremely soluble. The materials for the manufacture of calcium polysulphide are easily available; also it is on the market in both solutions and in powdered form, being used in agriculture as an animal dip and a fungicide. For these reasons more work was done with this reagent than with the others. The sulphydrate is likewise extremely soluble and better adapted for use than the normal sulphide.

RESULTS OF TESTS AND CONCLUSION FROM RESULTS.

The results of three tests with calcium polysulphide on the ore from the May Day mine of the Tintic district, in Utah, are shown in Table 43. At least 16 pounds per ton of ore seem to be necessary to get a satisfactory extraction of the lead, and recovery of the silver is very difficult. This latter point was observed in the tests with sodium sulphide on this ore, hence the inability to extract the silver in a satisfactory manner from this ore is not a peculiarity of calcium polysulphide.

TABLE 43.—*Results of sulphidizing ore from May Day mine with calcium polysulphide.*

[Assay of heading: Lead, 4.5 per cent; silver, 2.8 ounces per ton.]

Test No.	CaS ₂ used per ton of ore.	Time of treatment.	Oil used in flotation.	Concentrates.			
				Lead.		Silver.	
				In concentrates.	Extraction, percentage of total content of ore.	In concentrates.	Extraction, percentage of total content of ore.
	Pounds.	Hours.		Per cent.		Oz. p. ton.	
1	1.6	6	Coal creosote 1.2 pounds, and turpentine 0.4 pound per ton of ore.	12.5	32.2		
2	8.0	1.5	do.....	18.4	20.7		
3	16.0	3.6	do.....	26.1	73.0	11.56	48

Calcium polysulphide, although somewhat more sluggish, is otherwise as satisfactory as sodium sulphide for sulphidizing and flotation.

OTHER METHODS OF SULPHIDIZING.

SULPHUR VAPOR.

Oxidized lead ores can be sulphidized by treating the heated, pulverized ore with sulphur vapor, but the mechanical difficulties of applying the sulphur vapor caused the writers to discard this method as impracticable. Sulphur boils at 445° C., and in order to apply the vapor to the ore a temperature above this point must be maintained. Lead carbonate begins to break up into lead oxide and carbon dioxide at about 200° C. and is very easily reduced to metallic lead, so that there is little need of using sulphur vapor on such an ore at the temperatures that are required to prevent the sulphur vapor from condensing. The application to copper ores would not be attended with the same objections, as the various copper compounds have much higher melting points than lead sulphide and lead oxide. Elemental sulphur can be bought at coastal points in normal times for \$20 to \$25 per ton. It can also be cheaply distilled from pyrite at smelters or reduced from roaster gases by using an excess of reducing agent at a high temperature. Its commercial preparation for use in such a process need not be considered here.

SULPHURETED FLOTATION OIL.

The use of a sulphureted flotation oil is also possible theoretically. Much petroleum containing loosely combined sulphur is being refined by the use of copper oxide, which forms copper sulphide and leaves

the petroleum purified. Many of the flotation oils can be caused to take up sulphur and lose hydrogen, by distilling or boiling the oil with elemental sulphur. If the sulphur content in the oil is sufficient to form a film of lead sulphide over the lead carbonate particles, the flotation oil is evidently in the most advantageous position to "wet" the freshly formed artificial sulphide. In actual tests the writers were unable to get satisfactory extractions by this method. However, the results with sulphureted flotation oil and a sample that had previously been sulphidized with sodium sulphide were slightly better than those with nonsulphureted oil.

COLLOIDAL SULPHUR.

Attempts to sulphidize oxidized ores with colloidal sulphur, obtained by the interaction of hydrogen sulphide and sulphur dioxide, also failed, as colloidal sulphur does not sulphidize lead carbonate. If the ore has first been sulphidized with hydrogen sulphide, and the excess hydrogen sulphide is neutralized with sulphur dioxide to form colloidal sulphur, no froth is formed on the addition of a flotation oil, as all the froth seems to be "killed" by the sulphur dioxide or compounds formed in the action on the ore. Further, the writers have not found that colloidal sulphur can be successfully substituted for the flotation oil, although this point was given little attention after one or two failures.

"METALLIZING" THE LEAD.

Still another method of chemically altering an ore of lead in order to make it amenable to flotation is that of "metallizing" the lead. This was suggested by W. H. Coghill, who had tried the aluminium desulphurizing method, as practiced at the Nipissing mine, in Cobalt, Ontario, on an ore containing both silver and lead. He informed the writers that he had noted the formation of sponge lead during the tests.

In tests at the Salt Lake City station solutions of caustic soda and pieces of aluminium were agitated with the ore. The nascent hydrogen was found to desulphurize silver and lead, or reduce them to metallic form if they were present in oxidized forms. It was hoped that the metallized lead and silver could be floated. No difficulty in carrying out the metallizing operation with oxidized ores of lead was experienced, as sponge lead was visible, but this lead could not be floated. The presence of flotation oils of widely different types during the metallizing failed to improve the results, although it was possible to perceive small masses of sponge lead in the pulp. By rolling the pulp for three days in a bottle the sponge lead collected in small round balls which could be screened out of the pulp. The extraction of lead was high, but that of the silver was low.

As this method would be very expensive for lead alone, and was designed for the treatment of lead-silver ores that were refractory to other methods of treatment, the experiments were regarded as failures. One pound of aluminium can reduce 11 pounds of lead; hence the use of aluminium would be rather expensive.

Silicon or ferrosilicon will likewise react slowly with caustic soda solutions, reducing the lead compounds and also some of the silver. One pound of silicon will reduce about 17 pounds of lead. Silicon would be cheaper to use than aluminium, as it would reduce the metallizing cost per pound of lead to less than 1 cent. However, the method was considered of no value, as the extraction of silver was too low.

CONCLUSIONS AS TO RESULTS OF SULPHIDIZING TESTS.

The conclusions drawn from a comparison of the various methods of sulphidizing are as follows:

1. Hydrogen sulphide is the most active of the sulphidizing agents tested, but its poisonous and offensive character makes its use undesirable in ore-dressing mills.

2. Sodium sulphide and the other sulpho compounds of sodium are among the best alkaline sulphides for sulphidizing lead carbonate ores. The sulphhydrate of sodium is the most active, with the normal sulphide a close second. The cheapness of commercial normal sulphide and the small quantity required per ton of ore treated make it one of the most desirable agents for sulphidizing.

3. Polysulphide of calcium is the best calcium salt for commercial sulphidizing of lead carbonate ores, because it can be prepared cheaply from elemental sulphur and lime, and is readily soluble. It is less active in sulphidizing than the sodium compounds.

4. Other methods of sulphidizing, especially the use of elemental sulphur vapor, sulphuretted flotation oils, or colloidal sulphur, are not a success in flotation of the lead carbonates.

5. Metallizing a lead-silver ore by the use of caustic soda and aluminium (or ferrosilicon), followed by flotation, likewise failed, owing to inability to float the sponge lead and silver.

PRACTICAL APPLICATION OF RESULTS OF TESTS.

A number of alternative methods have been worked out for the treatment of low-grade oxidized ores of lead, and these methods vary widely in the conditions under which they can find application. If an ore is not suited, because of its physical or chemical properties, to treatment by a certain one of these methods, it is often possible to use some other method. The argentiferous lead carbonate ores require different treatment from the ores containing only lead.

LEACHING METHOD FOR LEAD CARBONATE ORES.

In the treatment of ores containing lead carbonate and no other valuable mineral, one of the simplest and cheapest methods of extracting the lead is that of leaching it out with acidified saturated brine. This method, however, can be used only for ores that contain small amounts of acid-soluble gangue material. If much alumina or lime is present, these constituents will consume the acid intended for the lead compounds, and hence one of the other methods will be required. In any event, the cost of the acid and the salt for getting the lead into solution has been found to be the greatest expense,

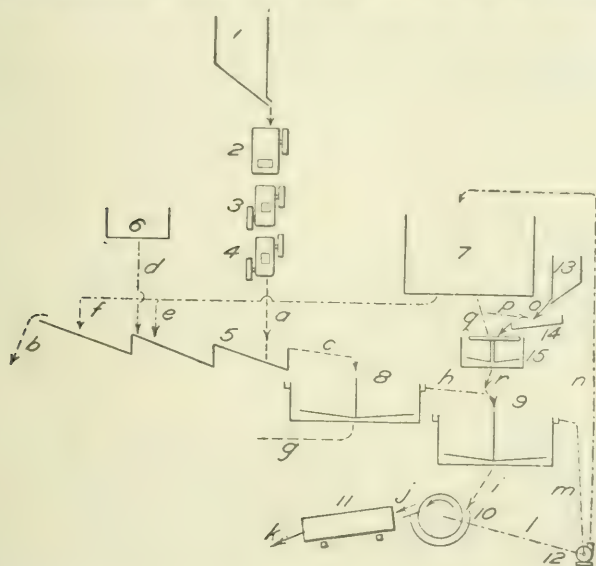


FIGURE 12.—Flow sheet of proposed mill for leaching oxidized ores of lead. 1, ore bin, capacity 100 tons per 24 hours; 2, crusher; 3, roughing rolls; 4, finishing rolls; 5, triple-washing Dorr classifier; 6, sulphuric acid storage; 7, brine storage; 8, Dorr thickener to remove slimes; 9, Dorr thickener to remove lead hydrate precipitate; 10, continuous vacuum filter; 11, revolving dryer; 12, pump; 13, lime-storage bin; 14, lime-slaking box; 15, Dorr agitator for making milk of lime. ——— Clear solutions. - - - - - Pulps. For significance of letters see table on page 109.

the cost of the lime or the electric current used for precipitation being small. An example of the calculated costs for treating one of the ores tested is shown on page 110. The estimates are based on the actual requirements as determined by tests of the ore.

FLOW-SHEET OF PROPOSED PLANT.

A flow sheet of a mill suitable for treating this ore is shown in figure 12.

CRUSHING.

In the proposed mill the crushing machinery consists of a crusher and two sets of rolls. Ore crushed to pass through a 10-mesh sieve was used in many of the tests, but probably ore considerably coarser

than 10 mesh could be successfully leached in the large plant. As most of these oxidized lead ores are soft and friable, it is probable that the crushing equipment could be simplified. Hence, all of the accessory apparatus, like elevators, are not shown.

LEACHING.

The tests have shown that less than a half-hour of contact of the acid-brine solution is necessary to get a 93 per cent extraction of the lead. Hence, it is proposed to use a number of Dorr classifiers in series for leaching the ore. Such machines were used during the life of the Butte and Duluth mill at Butte, Mont., with considerable satisfaction. They permit agitation of a coarse sand in contact with an acid solution, maple-wood rakes being used in their construction. The sand would pass through six of the classifiers in series in about 35 minutes. As shown in figure 12, most of the brine and all of the acid would be added near the head of the second classifier and the overflow into the first where the fresh ore enters. This would give a countercurrent of acid and ore. The third classifier would receive neutral brine for washing out the acid and lead-bearing brine. Water can not be used for washing, because saturated brine is necessary for good leaching of the lead. This condition is the main mechanical drawback to the proposed process.

SAND AND SALT.

The sand overflow, at *b* in the flow sheet, can be sent to the tailing heap or dropped into a sand tank to drain more thoroughly, in order to recover as much of the brine as possible. Where the cost of common salt is high it might pay to install apparatus for final washing of the sands with water and for evaporating part of the washings to saturation. A part of the waste water could be made into saturated solution direct by adding more salt to make up for mechanical losses. The moisture content of 1.65-mm. (10-mesh) sand can usually be reduced by draining to about 16 per cent. As the saturated solution contains 26.8 per cent sodium chloride, by weight, about 4 tons of that salt in 16 tons of solution would be retained by every 100 tons of sand. Hence 100 pounds of salt would be lost per ton of ore. The price of salt would not exceed \$5 per ton, except in the more isolated localities. The loss from salt in the tailing would rarely be more than 25 cents per ton of ore, and would usually be less, as such a leaching plant could probably get good rates from the salt manufacturers. In the vicinity of Great Salt Lake the cost of making salt from the lake brine is probably less than \$1.50 per ton, and the salt can be bought in large amounts for about \$2.

SLIME.

The slime, overflowing with the pregnant liquid, passes to a Dorr thickener, from which a product containing 60 per cent solids is run to waste. As many of the lead carbonate ores are known to produce about 20 per cent of slime when crushed to 10-mesh size, 100 tons of sand would yield 20 tons of slime containing 8 tons of liquid or 2 tons of salt—that is, the total salt loss would be 5 tons per 100 tons of ore. If the pregnant liquid entrained in the slime is washed out with barren brine, there should be recovered from 20 tons of slime 160 pounds of dissolved lead, which is worth 2.5 cents per pound at the smelter to the producer in the Rocky Mountain States. If the cost per day of operating a Dorr thickener is less than the value of the lead, it would be well to put in another thickener to recover the liquid. A large proportion of this liquid could also be recovered with a vacuum filter, which would have to be acid proof as the liquid is still slightly acid.

SOLUTION.

The clarified overflow solution from the Dorr thickener is passed into another Dorr thickener along with the proper amount of milk of lime, or rather of lime slaked in brine and suspended in brine. This neutralizes any remaining acid and precipitates the lead as a basic hydrate which settles out of solution fairly quickly. The precipitated solution is returned to the solution storage tank. The thickened sludge can be sent to an ordinary vacuum filter without further treatment and be washed free of solution with a small jet of water or of steam. The filter cake could be sent directly to a rotary drier. This filter need not be of acid-proof construction, as there is no acid left in the solution and no dissolved lead to be replaced by metal from metal parts of the filter.

CAPACITY OF PLANT AND COST OF OPERATION.

For those interested in the working of such a plant, the tonnage of every product at the different steps of the process is given in the following list. The letters in figure 12 refer to the letters in the following list and the accompanying weights of materials involved.

Significance of letters in figure 12.

- (a) 100 tons of ore, containing 10 per cent lead, of which 90 per cent can be extracted.
- (b) 67 tons of sand tailing, containing 1 per cent lead; 12 tons of solution, containing 3 tons of sodium chloride.
- (c) 18 tons of slime; 882 tons of solution, containing 236 tons of sodium chloride.
- (d) 8.5 tons of sulphuric acid, specific gravity 1.84.
- (e) 800 tons of solution.
- (f) 100 tons of solution.
- (g) 18 tons of slime tailing; 8 tons solution, containing 2 tons of sodium chloride.
- (h) 874 tons of overflow pregnant solution; 9 tons of lead.

- (i) 10.8 tons of precipitate; 10 tons of solution.
- (j) 10.8 tons of precipitate; 2 tons of moisture.
- (k) 10.8 tons of precipitate, containing 75 per cent lead, or 8.09 tons of lead.
- (l) 10 tons of solution.
- (m) 895 tons of solution.
- (n) 905 tons of solution.
- (o) 2.5 tons of lime (CaO).
- (p) 10 tons of solution for slaking lime.
- (q) 15 tons of solution for making up cream of slaked lime.
- (r) 25 tons of solution containing 2.5 tons of lime.

From this list the quantities of reagents used in every 24 hours on each 100 tons of ore can be calculated; they are as follows: Five tons of common salt; 8.5 tons of sulphuric acid (specific gravity, 1.84); 2.5 tons of lime.

In many places salt can be bought for less than \$5 per ton, delivered. In normal times sulphuric acid can be bought f. o. b. destination for not more than \$10 per ton, and, under favorable conditions, can be produced for less than \$5 per ton. Lime can be bought for about \$4 per ton. On this basis the total cost for chemicals would be about \$120 for 100 tons of ore, or \$1.20 per ton. The cost of chemicals will be about the same no matter how much lead is in the ore, as it takes a certain amount of these materials, regardless of the lead content. Careful management and the choosing of a proper location should reduce the costs somewhat. The costs of these materials might be reduced one-half if they were produced by the company extracting the lead, or to 65 cents per ton of ore treated.

In a small mill of the size considered, the costs of crushing the ore, leaching, filtering, drying precipitate, and pumping solutions need not exceed 75 cents per ton, and in a larger plant might be considerably less than 75 cents.

The total operating metallurgical costs would hence be about \$1.95 per ton of ore for a mill with a capacity of 100 tons a day, treating ore containing 10 per cent lead, with 50 per cent acid efficiency and 90 per cent extraction of the lead.

From 100 tons of ore 10.5 tons of precipitate containing about 75 per cent of lead as basic hydrate would be obtained. Most lead smelters will pay for 90 per cent of the lead in an ore sent to them and for an oxidized ore will make a base treatment charge of about \$2 per ton and pay a bonus of about 10 cents for every unit of lead above 25 per cent. On such a product as this precipitate, the bonus would amount to \$5 per ton. The cost of transportation from smelters in Utah to the lead markets and the cost of refining are deducted by the smelter, usually amounting to 1.25 cents per pound. Hence, with lead at 4 cents per pound, this precipitate should bring about \$40.10 per ton, or \$421 for 10.5 tons, the quantity obtained from 100 tons of ore.

The first cost of a plant that should last 3 to 5 years, treating 90,000 to 150,000 tons of ore in its life, is roughly estimated at \$25,000. To offset this, a depreciation charge of 15 to 25 cents per ton of ore treated should be made. Interest on the investment at 10 per cent would amount to \$2,500 per year, or \$.35 cents per ton of ore, 300 working-days per year being assumed.

The total cost of mining most carbonate ores will average \$3 to \$2.50 per ton, or probably a little less.

The cost of transporting the ore to the mill will vary according to conditions and may be practically nothing or several dollars per ton.

Summing up the costs of such a plant, we have:

Estimated cost per ton of ore treated for plant treating oxidized ores.

	Cost per ton of ore treated.
Mining.....	\$2.00
Metallurgical.....	1.95
Depreciation.....	.20
Interest.....	.08
Total.....	4.23

MATERIAL THAT COULD BE TREATED.

The value of the lead precipitate extracted from ore containing 10 per cent lead is \$4.21 per ton of ore treated. Hence, unless the cost of mining and treatment can be reduced below the figure given, such ore could not be worked at a profit. An increase in the grade of ore would increase the cost for chemicals 17.7 cents for every increase of 1 per cent in lead content. The value of 1 per cent of lead, added to the final precipitate and allowing only 90 per cent extraction, would be 37.9 cents. Hence, an ore containing 11 per cent lead would yield a profit of 36 cents, a 12 per cent ore, 72 cents per ton, and so on. In many localities, the cost of chemicals would be much less than \$1.20 per ton of ore, the figure used in the computations. As was pointed out, the cost of these materials might easily be cut to 65 cents per ton, and the other milling costs to as low as 35 cents for such a small mill. This would make the total cost 95 cents less and permit a profit of 93 cents per ton on 10 per cent lead ore. If the mining costs could be cut to \$1 per ton, the lowest grade of ore yielding a profit would be ore containing about 6 per cent lead.

Further, in treating some ores the acid efficiency would be much higher than the 50 per cent when for every pound of acid that does useful work another pound is lost. This would permit a considerable saving in acid, the cost of which makes at least half of the cost for reagents. Such ores often consist largely of quartz and other insoluble minerals.

Briefly, it can be said that with lead selling at 4 cents a pound, treatment of the low-grade lead carbonate ores in the Rocky Mountain

States in an acid-brine leaching plant is practicable. A 100-ton plant would cost \$25,000 and should have a life of at least three years, recovering 90 per cent of the lead and yielding a basic hydrate precipitate of lead, which could be marketed to advantage. The lower limit of the grade of ore that can be profitably treated would be 6 to 11 per cent lead, according to geographical and other conditions.

ESSENTIALS REQUIRED BY PROCESS.

The requirements for the success of such a process are:

1. The ore shall contain lead carbonate (or sulphate) and a more or less insoluble gangue, so that an acid efficiency of 50 per cent or more can be obtained.
2. Salt, sulphuric acid, and to a minor extent, lime, will have to be available at reasonably low figures—\$1.50 to \$5 per ton for salt, \$5 to \$10 for acid, and \$3 to \$5 for lime.
3. The extraction of precious metals is small, with the exception of silver present as the chloride, which is also soluble in the brine. Hence, most ores containing precious metals can not be treated by this process.

ALTERNATIVE LEACHING METHODS.

The method outlined for leaching lead from its carbonate ores needs modification under certain conditions.

CONVERTING PRECIPITATE TO METALLIC LEAD.

If the cost of transporting the lead precipitate to the smelter is high, or if the precipitate has to be transported over difficult roads in conveyances from which some of the finely divided precipitate may be lost, it may pay to convert the precipitate into bars of metallic lead at the leaching plant. This is easily accomplished with precipitate containing 75 per cent lead, as the lead hydrate can be calcined to oxide and the oxide reduced with little more than the theoretical amount of reducing agent at a low temperature, in simple apparatus. That is, one ton of precipitate will require about 125 pounds of charcoal for its reduction, and enough fuel to bring the charge to about a dull red heat. The impurities in the precipitate are crossed off.

The value of the lead obtained from 100 tons of the 10 per cent ore as mentioned on page 110, would be \$495, at 4 cents per pound, and the precipitate containing 75 per cent lead would be worth \$421. The difference of \$74 can be balanced against the cost of reduction of the 10.8 tons of precipitate, thus permitting a charge of \$6.85 per ton for reducing the precipitate. If the precipitate can be reduced for less than \$6.85, as seems very likely with such easily reducible material, it would pay to add this step to the milling process. The saving in freight to the smelter is also to be considered.

RECOVERY OF SILVER.

If some of the silver in a lead-silver ore is soluble in the brine and tends to be precipitated with the lead hydrate, the reduction of the latter to metal would give base bullion instead of pig lead. In order to obtain most of the lead in a desilverized condition, it might be best under such conditions to remove the silver by precipitation on scrap iron or on sponge lead before the lead is precipitated. Scrap iron is a good precipitant of silver and only a small proportion of the lead will accompany it. In this way all of the silver in solution can be recovered and melted down into a small quantity of base bullion containing some lead. The rest of the lead could then be melted down free of silver.

ELECTROLYTIC PRECIPITATION.

Many ores have too large a content of soluble impurities to permit the preparation of a lead-hydrate precipitate of sufficiently high grade. Zinc and iron are the common impurities of this character in the materials tested. A small proportion of manganese was found in some of the precipitates, and an ore high in that metal might possibly leave too much of it in the precipitate. Lead can be precipitated from the solution by electrolysis, the method having been explained previously. Electrolytic equipment is always expensive and adds much to the first cost and the overhead costs of a plant, so that mining and treatment would have to be on a large scale to make the use of this method of precipitation profitable. If zinc is the principal impurity, it could be permitted to build up in the brine until it began to affect the solubility of the lead, and then be precipitated as hydrate with lime, as the lead is. Equipment for electrolytic precipitation, including transformers, motor-generator set, buss bars, and electrolytic tanks, having a capacity of 10 tons of lead per day would cost about \$16,000 and should have a life of 10 years. The power consumed is about 1 kilowatt-hour for 5 to 15 pounds of lead, and constitutes a small part of the total electrolytic costs.

In electrolytic precipitation the lead is deposited as metallic sponge that is easily melted and is an excellent precipitant for silver. A small part of this sponge could be used for separating out the silver before electrolysis of the lead. Only a small amount of base bullion, requiring desilverization, would be formed, whereas the bulk of the lead would be ready for market, as shown by the analyses of the bars melted down in the laboratory. The only drawback to the electrolytic method used in the tests was that soluble iron anodes were used in order to reduce the power consumption. The weight of iron used up is equivalent to the lime used in the lime-precipitation method and costs much more.

ELECTROLYTIC AND LIME-PRECIPTATIONS METHODS COMPARED.

Comparing the requirements of these two methods of making metallic lead at the leaching plant, we find:

1. Lime precipitation followed by reduction of the precipitate requires an ore relatively free from soluble zinc and manganese minerals, whereas by the electrolytic method very impure solutions can be handled.

2. The weight of lime used in the first method is equivalent to the weight of scrap iron used in the other. The cost of these two materials must be investigated, in order to compare the two methods. Usually lime can be bought more cheaply than scrap iron.

3. In the first method coal is used for reduction of the lead to metal, and in the other, electric current. In each method coal is used for heating the melting pots to permit melting and casting the lead and drossing off any impurities. In either method the amount of coal or of electric current utilized is such a small part of the total cost that this item is of minor importance.

4. Although not definitely determined, it is probable that the electrolytically precipitated material will yield lead of higher quality, as a single analysis of such lead showed a very small content of impurities.

5. The installation and overhead costs of a plant using the electrolytic precipitation method would be higher than for a similar plant using precipitation with lime followed by reduction with carbon.

6. In either plant the advisability of reducing the precipitate to metallic lead before shipping depends on the proximity to a lead smelter and the price the smelter is willing to pay for the precipitate.

7. Silver in the leaching solution is more easily taken care of by the electrolytic process, as some of the sponge lead precipitated by electrolysis can be used to precipitate the silver before the solution enters the electrolytic tank. However, this point is only of minor importance.

VOLATILIZATION METHOD.

As pointed out already, some deposits of lead carbonate ores are too far from a suitable water supply for treatment by either leaching or flotation methods to be practicable. For these ores volatilization of the lead from the ore, as chloride, seems to be better adapted. For purposes of comparison with the other processes described herein, an attempt is made to evaluate costs for this process.

PROPOSED PLANT FOR USE OF METHOD.

For a 10 per cent lead ore 7.5 per cent sodium chloride and 5 per cent coal dust are required for volatilization, or 150 pounds of salt and 100 pounds of coal dust per ton of ore. For fluxing the lead

chloride precipitate about 50 pounds of lime and 10 pounds of coal dust are required, also fuel is needed for heating the melting pot or furnace used. This should yield 180 pounds of lead, or a 90 per cent extraction.

For a 100-ton mill, the cost of salt, coal dust, and lime would be approximately as follows:

	Cost per 100 tons of ore.
7.5 tons salt, at \$5.....	\$37.50
7.0 tons coal dust, at \$3	21.00
2.5 tons lime, at \$4.....	10.00
Total.....	68.00

The operation of such a mill, as can be seen in figure 13, is very simple. The ore after being crushed in the dry state to about 10-mesh size, is mixed with the proper proportions of salt and coal dust or other fuel, and sufficiently moistened with water to ball slightly. This mixture is fed onto the grates of a Dwight-Lloyd sintering machine, or similar down-draft roaster. The lead volatilizes as lead chloride and the sintered residue is discharged to the tailing dump. The gases pass through an electrical precipitator, leaving their suspended burden of lead chloride. The precipitated lead chloride is mixed with lime and a small amount of coal dust and melted down in a pot to metallic lead and calcium chloride slag. Any lead chloride volatilizing in the melting operation is caught by the electrical precipitator again.

The list of machines and products, as represented on the sketch shown in figure 13, follows:

List of parts and products shown in figure 13.

1. Crushing department, containing jaw crusher, rolls, etc., or equivalent equipment.
 2. Distributing belt.
 3. Ore bin.
 4. Coal-dust bin.
 5. Salt bin.
 6. Mixing belt.
 7. Christensen mixer.
 8. Mixed-ore bin.
 9. Dwight-Lloyd or equivalent down-draft roaster.
 10. Suction fan or blower, 24,000 cubic feet per minute.
 11. Electrical precipitator.
 12. Lead-chloride bin.
 13. Lime bin.
 14. Melting pot or small reverberatory reduction furnace.
 15. Water tank.
- (a) 100 tons of ore, containing 10 per cent Pb, per day.
 (b) 100 tons of ore, containing 10 per cent Pb, passing a 10-mesh screen.
 (c) Coal, passing 10-mesh screen, 5 tons per day.
 (d) Salt, 7.5 tons, granulated.

- (c) Water, 6 tons.
- (f) Sintered tailing, 85 tons.
- (g) Gases from electrical precipitator, 24,000 cubic feet per minute.
- (h) Lead chloride fume, 12.85 tons containing 70 per cent Pb.
- (i) Lime, equivalent to 2.5 tons CaO.
- (j) Bars of lead, 9 tons.
- (k) Calcium chloride, 5 tons.

The 5 tons of calcium chloride obtained from melting the lead chloride is equivalent to 2.6 tons of sodium chloride and can be used in the mix that passes to the down-draft roaster 9. This would

reduce the cost for sodium chloride \$12 and make the total costs for reagents \$56 instead of \$68.

The other operating costs per ton of ore treated will be approximately as follows: Crushing, \$0.20; mixing, \$0.05; roasting and precipitation, \$0.35; melting and casting, \$0.25; total, \$0.90.

The first cost of such a plant is estimated at about \$50,000 and its life as five years. Hence a depreciation charge of 33 cents per ton of ore treated in the plant during its life should be made. Interest at 10 per cent per annum on the investment amounts to 16 cents per ton of ore treated.

Summing up, the metallurgical costs per ton of ore would be: Chemicals \$0.56, and operation of plant \$0.90, total \$1.46.

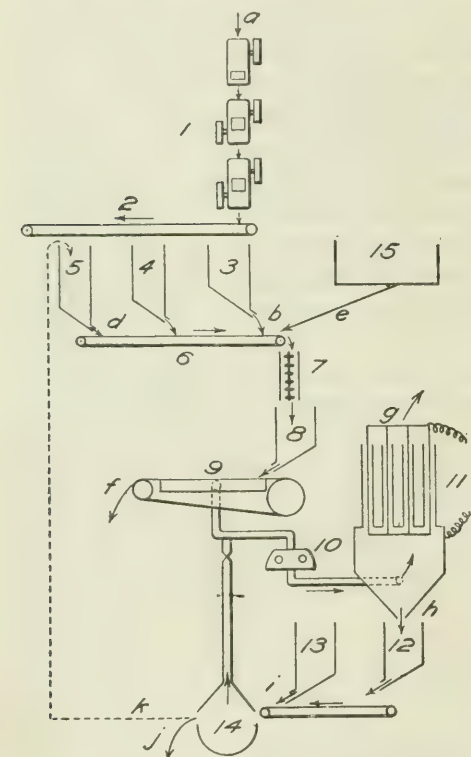


FIGURE 13.—Flow sheet of mill for volatilization of lead. For sign figures of numbers and letters, see table on opposite page.

At \$2.50 per ton for mining, the entire cost per ton of ore mined and treated would be as follows: Mining, \$2.50; metallurgical treatment, \$1.46; overhead expense, \$0.49, or a total cost of \$4.45 per ton of ore treated.

The value of the 180 pounds of lead recovered, at 2.75 cents net (4 cents gross) per pound, would be \$4.95, yielding a profit of \$0.50 per ton of ore treated, even under the somewhat high costs assumed. The lead is very pure and does not need to be refined or desilverized,

so the deduction of 1.25 cents per pound of lead from the market quotation of 4 cents is somewhat excessive. With each increase of 0.25 cent per pound in the price of lead, the corresponding saving on a ton of 10 per cent ore would be 45 cents.

The volatilization process seems to have some advantage over the leaching process, but requires a greater initial outlay and is hence less adapted to the treatment of small deposits of ore.

If material from a mill dump is to be treated, the cost of recovering such material would be much less than mining, and a dump of 10 per cent lead content would yield a handsome profit on the same basis of operation. With a cost of \$2 per ton, and 90 per cent recovery, a 10 per cent lead ore in normal times would yield a profit of \$2.95 per ton in a 100-ton mill working 300 days per year, and the return on the investment in one year would be 177 per cent.

REQUIREMENTS COMPARED WITH OTHER METHODS.

To summarize, the requirements of this process in comparison with the two leaching methods before described are as follows:

1. The volatilization process, when a down-draft sintering machine is used as a roaster, is adapted to the treatment of an ore containing only lead. Silver, zinc, copper, and other metals are not extracted from an ore by this method.

2. As the process is an igneous one, little water is required, and the process can be used in arid regions where leaching or flotation would require too much water.

3. The operation of a plant using this process is simple and almost automatic, the labor required being largely for the supervision of machinery, with the exception of melting the lead chloride.

4. Very fine grinding is not necessary, although the ore must be ground dry. Modern dry-grinding ball mills are ideal for this purpose.

5. The reagents necessary for the process—salt, lime, and coal—are standard articles of commerce and easily available in practically every mining district.

6. The final product is metallic lead ready for market, hence transportation costs are reduced to a minimum. The cars used for bringing salt, lime, and coal to the mill can be loaded out from the mill, which should permit better transportation rates.

7. The machinery used is all standard types for sale on the market, except the Christensen mixer, which is of simple construction, being made from a piece of iron pipe and a piece of shafting to which are attached short cross bars.

8. The first costs of such a plant would be higher than those of a leaching plant, but the total cost of operation would be lower.

ROASTING AND LEACHING.

The chloridizing-leaching process, as developed in Utah by N. C. Christensen and T. P. Holt, at first working together and later independently, consists of roasting the ore with salt in the presence of fuel and a sulphide for obtaining a chloridizing atmosphere. The ore, after being roasted at slightly above 600° C., is leached with brine that is about 75 to 80 per cent saturated with salt and is acidified with sulphuric acid.

Christensen used a down-draft roaster and Holt used a shaft roaster with up-draft through the bed of ore. The temperature was not allowed to rise high enough to volatilize much lead, although it is impossible to prevent considerable volatilization of lead at any roasting temperature. The modification of using fully saturated brine will permit the process being applied to the treatment of argentiferous lead carbonate ores, with recovery of copper and gold as well. The combination plant for volatilization and leaching mentioned below could be used for this purpose, by further reducing the draft so that lower roasting temperatures could be used. In that event the electrical precipitator would be rendered useless, and instead the gases could be passed through an acid tower and scrubbed with mill solution in order to catch any acid formed by the roasting.

The process, as outlined, is well known, except for the fact that the use of saturated brine will permit the recovery of lead. The authors do not believe that the process is so well adapted to such ores as partial volatilization, followed by leaching, for the reason that greater volumes of brine are necessary to dissolve all of the lead from the ore. The total costs are known to be about \$2.50 to \$3 per ton of ore.

VOLATILIZATION AND LEACHING.

The volatilization process described not being adapted to the treatment of argentiferous lead carbonate ores, a modification of the plant shown in figure 9, page 59, is necessary for recovering the silver. Such a plant would be similar to the plant sketched in figure 9, except that the calcines from the down-draft roaster would be put through a leaching plant resembling that shown in figure 8, page 48. The roaster would have to be set for lower draft and the ore not sintered. Only 75 per cent of the total lead content could be extracted by volatilization; the rest could be leached with a brine solution along with the chloridized silver, copper, and gold.

This modified process has the following characteristics:

1. A small leaching plant can be used for extraction of the residual lead, because a smaller volume of brine will be necessary for extracting the residual lead than when leaching the raw ore.

2. The silver, gold, and copper content of a mixed carbonate ore can be recovered together with the remaining lead.

3. The roasting operation breaks up ore colloids and leaves a product that can be more easily leached, and all slime troubles are eliminated.

4. The operation of both volatilization and leaching plants causes higher costs, which are only justifiable on account of the recovery of metals other than the lead.

5. The addition of sulphides, like pyrite, to the roast in order to help chloridizing of the silver and the copper is an added expense.

6. The necessity of leaching makes this process poorly adapted to arid regions.

VOLATILIZATION OF LEAD ORES CONTAINING PRECIOUS METALS.

All of the lead, silver, gold, and copper content could be extracted from an oxidized ore by mixing the ore with salt and by volatilization roasting at about 900°C . for a sufficient length of time. The down-draft sintering machine and similar roasters are not applicable for this purpose, because the duration of the roast is too short. Reverberatory or kiln roasters capable of giving the proper temperature for one to two hours would be required. Otherwise the flow sheet of the plant used would be practically similar to that shown in figure 13, page 116. The gold and silver being extracted with the lead and later reduced with it would necessitate sending the pig lead to a refinery; so that the whole \$1.25 for each 100 pounds of lead would have to be deducted for shipping and refining charges. The roasting would doubtless be more expensive and would consume more fuel, but otherwise the total costs would be about the same as those previously given. Oxidized ores containing 3 to 8 per cent lead and 1 to 8 ounces of silver, in siliceous or limestone gangue, are fairly common in the intermountain region, and are rarely mined, even for gravity concentration.

The advantages of this process, in comparison with the others mentioned herein, when considered from a commercial standpoint are:

1. Salt, lime, and fuel are the only materials needed, and these are readily obtainable. Special materials like sulphuric acid are not needed, and hence the process is not dependent on a cheap supply of acid.

2. High extractions of all the desirable metals are possible, and almost any type of gangue is permissible provided that sintering or slagging does not take place at too low a temperature.

3. The process is adapted for use in arid regions and at plants at a considerable distance from a railroad.

4. The apparatus necessary is simple and inexpensive, and can be so designed that a minimum of labor is necessary. This tends toward low first costs and low operating costs and makes possible the treatment of relatively small deposits.

5. This process is considered by the writers as one of the most important in its chance of future adoption.

6. All the operations proposed as steps of this process are at present in use in commercial plants or have been tested on a large scale in times past.

SULPHIDIZING AND FLOTATION.

Flotation of the carbonate ores of lead, although the method seems simple and adapted to be introduced in ordinary concentration mills, yields only a concentrate that must be shipped to smelters for further treatment. However, the most important question, as regards the application of this method to such ores, is the cost of treatment. Hence an attempt to set forth the costs of this process follows:

COSTS OF PROCESS.

Most of the ores treated in the Salt Lake City laboratory of the Bureau of Mines have required about 20 pounds of sodium sulphide per ton of ore, for the most successful treatment. The price of sodium sulphide in normal times is about 2 cents per pound, but the war in Europe has caused prices to rise to 4 and 5 cents per pound.

With the exception of the cost of sulphidizing reagents, costs of operation of a sulphidizing and flotation mill are about the same as those in ordinary flotation mills. For a plant treating 100 tons a day of ore containing 10 per cent lead the costs are estimated to be as follows:

	Cost per ton of ore.
Sodium sulphide, 20 pounds, at 3 cents per pound.....	\$0.60
Milling.....	.75
Total.....	1.35

If it be assumed that an 80 per cent extraction of the lead and a concentrate containing 50 per cent lead is obtained, the daily output would be 18 tons of concentrate. In buying such concentrate the lead smelters will pay for 90 per cent of the lead content at 1.25 cents per pound of lead less than the prevailing market price, St. Louis quotations, whereas the charge for smelting lead ore or concentrate is \$2 per ton for 25 per cent lead content with a bonus of 10 cents per ton unit for every unit of lead over 25 per cent. Hence, the 50

per cent concentrate would be credited with 50 cents per ton net after treatment charges have been deducted, provided the "insoluble" and the iron contents balance to make a self-fluxing ore. Most of the concentrates obtained in the tests have contained an excess of "insoluble" over iron. As the penalty for excess of "insoluble" is 10 cents per unit the probability is that, all things being considered, the bonus would vanish and the ore would be treated by the smelter free of charge. The ability of smelters to treat flotation concentrates has not yet been satisfactorily settled, and it may be that in the near future the smelters will make a charge for sintering and otherwise preparing the concentrates.

The cost of transportation to the smelter will usually average several dollars per ton of concentrate for districts far enough from a smelter to make flotation of the ore before smelting profitable. If the mill is at a distance from the railroad the cost of hauling the concentrate to the railroad becomes a serious item.

With lead selling at 4 cents per pound, the concentrate will be worth about \$25 per ton delivered at the smelter, or \$450 for 18 tons, the daily output of the mill. As the original mill feed was estimated at 100 tons of ore daily, from each ton of ore \$4.50 worth of concentrate would be recovered.

A conservative estimate of the cost of mining the average lead carbonate ore is the figure used in previous calculations, \$2.50 per ton.

Summing up the operating costs, we have:

	Cost per ton of ore.
Mining.....	\$2.50
Milling.....	1.35
Transportation of concentrate to smelter.....	.54
Total.....	4.39

The first cost of such a plant will be in the neighborhood of \$20,000. With a life of five years, the depreciation would be \$4,000 a year, and interest at 10 per cent would amount to \$2,000 a year. The total overhead charge is thus \$6,000 a year, or 20 cents per ton of ore treated. Hence, the total cost of treating this type of ore by the flotation process would be \$4.69 per ton, whereas the value of the lead concentrate is only \$4.50 per ton of ore.

For treating material that has already been mined, such as mine and mill dumps, with no mining cost to be charged against the method, the cost of treatment would be low compared to that of some of the other methods. The costs of milling and of the transportation of concentrate amounts to \$1.89 per ton of material. This would leave \$2.61 per ton profit. Furthermore, many old mill

dumps that do not require grinding would permit of a less expensive mill than the one calculated on.

It must also be remembered that when precious metals are associated with the lead, as there usually are in the ores of the mountain States, the value of the precious metals may make the concentrates sufficiently valuable to permit concentration of the same grade of mine ore at a profit.

CONCLUSIONS AS TO FLOTATION OF LEAD CARBONATE ORES.

Summarizing, the principal points about flotation of the lead carbonate ores as compared to other methods are as follows:

1. Not all ores can be treated by sulphide filming and flotation, but for those that do yield to such treatment the process seems to have commercial possibilities.
2. Equipment similar to that necessary in a flotation mill of the ordinary type can be used, with the possible addition of a thickener or a mechanical agitator for applying the sulphidizing solution.
3. In addition to the ordinary cost of milling there is to be considered the cost of such a reagent as sodium sulphide, which will be 10 to 75 cents per ton of ore.
4. In ores containing silver and gold in addition to the lead, the gold and the silver are often associated with the lead minerals, and consequently such ores are amenable to treatment by this method.
5. The greatest obstacle to the commercial success of this method is the cost of shipping the concentrate to the smelter. The methods that produce either metallic lead or lead hydrate would have much lower transportation and refining charges.
6. The method requires an ample supply of water and hence is not adapted to conditions in arid regions.
7. For working mine and mill dumps a 10 per cent lead content would be highly profitable at normal prices, and considerably lower grades could be treated by this method.
8. The first cost of a plant for flotation of lead ores is cheaper than that of any other type considered, with the exception of a gravity concentration mill. It is assumed that only ores not amenable to gravity concentration are in question.

SULPHIDE ORES OF LEAD.^a

PROBLEMS INVESTIGATED BY THE BUREAU OF MINES.

Two major problems in the treatment of the sulphide ores of lead were given attention when the work on such ores at the Salt Lake City station of the Bureau of Mines was begun.

RECOVERY OF GALENA FROM SLIMES.

The first problem is that of recovering galena from the slimes of concentration mills. This mineral is often so friable that it slimes badly when crushed for concentration, consequently the slime losses were formerly serious. It was not uncommon, only a few years ago, to find concentration mills losing 1 pound of lead for every 2 saved in concentrates.

This condition of affairs has long been recognized. The Bunker Hill & Sullivan Mining & Concentrating Co., of Kellogg, Idaho, two or three years ago started an experimental plant for determining the best way to recover these losses. The Malm process of dry chloridizing was tried and failed, partly because the ore chiefly contained lead and had only a small content of zinc, whereas the Malm process is designed primarily for ores with a high zinc content. It was noticed that during the treatment of the ore with chlorine and in its subsequent roasting the lead was converted to sulphate of lead and that this lead sulphate was soluble in strong salt solutions. This was said to be the discovery of R. S. Handy, the mill superintendent, and was patented by him.^b

The discovery, however, is not new, as it formed the basis of a process used in 1854 by Bequerel,^c of the Academy of Science of Paris. Bequerel claims that he worked with over 20,000 pounds of ore from Mexico, South America, and the Erzgebirge and the Altai Mountains. His method consisted in either a chloridizing or a sulphating roast of the ore to render the various metals soluble in brine, followed by a brine leach. The soluble compounds were said to be silver chloride and lead sulphate. The solution obtained was electrolyzed. The method was not a commercial success, because the only available source of electric current in those days was from the primary cell, a source of power that was entirely too expensive.

^a Experimenters: C. L. Larson, M. J. Udy, and H. C. No. 44.

^b Handy, R. S., Process of treating ores; U. S. patent 1185702, Oct. 28, 1914.

^c Bequerel, M. M., etc., *Traité d'électricité et de magnétisme*. Paris, 1855. Vol. 2, pp. 276-446.

The fact that galena can be caused to roast largely to lead sulphate and that the lead sulphate is soluble in strong brine, although known long ago, has never been utilized profitably and has been rediscovered a number of times, as at Kellogg. The writers found this old process being tested in the laboratory at Kellogg, and later found that it had been utilized more or less by N. C. Christensen in his millwork at Silver City, Utah, where a chloridizing roast was being given to oxidized or semioxidized ores, followed by a brine leach. In Christensen's work the brines used were not saturated solutions and hence could not exert the same solvent effect as saturated brine. Christensen, however, recognized the value of concentrated solutions of brine for obtaining better extractions of lead.

The work at the Salt Lake City station has been a further investigation and development of these discoveries. The Bunker Hill & Sullivan Co. has likewise continued the development of this method, and has cooperated with the Bureau of Mines to a considerable extent. Some of the data herein presented were obtained in experiments at the bureau's station and some were obtained from the operation of a semicommercial plant by that company. In the meantime the advent of flotation solved the problem of the slime losses for the company and made the further development by it of a hydrometallurgical process less necessary. Smelter problems, however, brought up the question as to the best method of treating all the ore of the mine, whether by concentration and smelting or by hydrometallurgical treatment of both the shipping and milling ore on the ground. Necessity for immediate action caused the company to build a smelter to smelt the concentrate from its mills, although the development of the details of the lead leaching process was not abandoned. A comparison of the two alternative methods will be found on pages — to —.

TREATMENT OF COMPLEX SULPHIDES.

The second major problem involving sulphide ores of lead is that of lead contained in the so-called "complex" sulphides. Complex sulphides are usually rendered such by the presence of zinc sulphide with the lead sulphide, and iron and copper sulphides often accompany them. If the various sulphide minerals are present in the ore in coarse grains, there is usually little difficulty in separating them from each other by modern methods of ore dressing. However, there are many ores in which the mineral grains are so small that no ordinary ore-dressing process will separate them. These complex sulphide ores may often have a large content of lead, zinc, silver, gold, and copper, but the impossibility of separating the minerals from each other makes the ore of little value or even worthless.

Hence the feasibility of roasting and leaching these ores in order to remove the lead by the acid-brine leaching process, or by the subli-

mation process, was investigated. The particular types of ore to which these methods should apply are as follows:

1. Zinc sulphide concentrates containing some (up to 10 per cent) lead.
2. Zinc-lead sulphide ores not amenable to concentration.
3. Pyrite containing lead, in localities too far from a lead smelter to permit shipment.

The methods that have been tested in the bureau's laboratory for the solution of these problems are:

1. Differential flotation.
2. Roasting and acid-brine leaching.
3. Sublimation and acid-brine leaching.

SIMPLE SULPHIDE ORES OF LEAD.

In taking up the problem of the treatment of the simple sulphide ores of lead, as previously outlined, the application of flotation to prevent slime losses of galena during concentration need hardly be explained. At the inception of the work at the Salt Lake City station the value of flotation for such ores was not as apparent as it now is and recoveries were often low. Since the introduction of flotation, the recoveries of lead sulphide from disseminated ores have been raised to as much as 95 per cent. For a concentration process such recoveries are remarkably high. However, hydrometallurgical processes, in which solutions have a chance to penetrate to the very center of the small particles of ore, are usually capable of very high extractions, and usually have the added advantage of making metal direct. On that account the methods described below are still of considerable interest, as they encourage the hope of a lower cost of production.

PRELIMINARY LEACHING TESTS AT BUNKER HILL & SULLIVAN PLANT.

When it was discovered that saturated brine would dissolve lead sulphate the Bunker Hill & Sullivan Co. started a series of cyclic experiments in which the slime tailing, after being mixed with salt, was roasted and then leached with brine. The object of a chloridizing roast was to convert the silver associated with the lead to the form of silver chloride, which is soluble in brine. By using the same brine repeatedly on a series of more than 30 roasts, and precipitating the lead after each leach by adding sufficient lime, it was found that apparently the solution did not deteriorate, and that satisfactory extractions of the lead, with 40 to 70 per cent of the silver, were possible, depending on the success of the roast. In the leaching of

each roast enough wash water, or weak brine, was used to make up for losses of solution in the tailing and for accidental losses in the laboratory. This introduction of fresh weak solution each time recovered a great deal of the salt, and also was later shown to be the cause of the solution not fouling during the cyclic work. The roasts were made in a piece of 4-inch iron pipe that was laid across a blacksmith's forge and slowly rotated by means of a worm gear attached to a small motor. Roasts conducted at temperatures varying from 400° to 700° C. permitted good extractions of lead in nearly all tests, whereas the best extractions of silver were in roasts conducted at temperatures of 600° to 700° C. A roast lasting about one hour at these latter temperatures, and in such a roaster, was sufficient. All of the leaching was with hot brine. This work was later checked by use of a test plant capable of treating a maximum of 10 tons of ore per day. After all leaks had been stopped in the test plant it developed that the solutions gradually fouled by the building up of sodium sulphate and other compounds and by the falling off of sodium chloride in the solution. Occasionally it was noticed that if hot solution from the leaching tanks or filters was permitted to cool, a considerable amount of lead chloride would crystallize out on cooling. Some tests were made to determine whether it might be possible to obtain lead chloride in such a way and later treat it for the lead content. It was found that it was not always possible to obtain solutions sufficiently saturated with lead chloride to yield crystals on cooling. The method of lime precipitation seemed to be better adapted for consistent recovery after the leaching of the lead from the brine solution.

LEACHING TESTS BY BUREAU OF MINES.

At this point the bureau took up the further study of the process, cooperating with the Bunker Hill & Sullivan Co. by exchange of data and by occasional visits. It was decided to try to avoid the use of heated solutions if cold solutions could be used just as well. The use of cold solutions was found to be feasible. Also it was determined that a ratio of solution to ore such that each liter of solution would contain 10 grams of lead was satisfactory. As the material being tested contained about 7 per cent lead, a pulp ratio of about 7:1 would have to be used. Such a ratio in leaching is not excessive. The heating of solution is expensive, and as good extractions of lead by the use of not too large amounts of cold brine were possible, all further work was with cold solutions.

It was determined that the lead could be easily converted to a form soluble in the brine (either lead sulphate or chloride) by roasting, and that satisfactory extractions can be obtained by roasting

through a wide range of temperatures. Sodium sulphate is formed by the action of the sodium chloride on the lead compounds. When the calcine from a chloridizing roast is moistened the sodium sulphate immediately dissolves and reacts with any lead chloride present to form lead sulphate. Hence it was impossible to determine how much lead chloride and how much lead sulphate were formed in the roast, as all the lead soluble in neutral brine would be converted to lead sulphate in the attempt to leach out the lead chloride with hot water. However, it was found that 80 to 99 per cent of the lead in the ore was converted to a form soluble in brine in the roasts as performed at the Bunker Hill & Sullivan experimental plant. It was also determined that about 10 per cent of the lead in the ore was volatilized in such roasting. The consumption of salt (NaCl) in roasting was also determined and was found to be 20 to 40 pounds per ton of the material tested. Further, although the Bunker Hill & Sullivan slime tailing contained only 4 per cent of sulphur, it could be roasted with no additional fuel in a blast roaster of the Holt-Dern type.

Two series of cyclic experiments with cold leaching were carried out in the laboratory at the Salt Lake City station with a number of samples of calcines from the roasters of the company's experimental plant. In one of these series of cycles the calcines were leached with brine to remove the lead, and then lime was added to the leach to precipitate hydrated oxide of lead and regenerate the solution for further leaching. The sodium sulphate in the solution was decomposed by adding sufficient calcium chloride to the brine at the beginning of the leach to react with all soluble sulphates and form calcium sulphate, which is relatively insoluble and is left behind in the tailing.

In the other series of cyclic leaches the use of calcium chloride for precipitation of sulphates was omitted in order to determine the effect of permitting the sulphates to build up in the solution. Over 30 complete cycles were followed through in each series.

LEACHING WITH ADDITION OF CALCIUM CHLORIDE TO PRECIPITATE SULPHATES.

The results of the first series of tests are shown in Table 44 following. In the first six cycles no calcium chloride was added, in order to determine whether any noticeable effect would take place after the addition of calcium chloride was commenced.

In cycles 7 to 19, inclusive, enough calcium chloride was added to precipitate the sulphates in the solution before application to the ore. In Nos. 20 to 30 the solution was permitted to become slightly

basic with lime during precipitation and was then neutralized with hydrochloric acid. As a result calcium chloride was formed in the leach liquor. In test 21 the solution contained 0.35 per cent of CaO (the Ca being present as calcium chloride or as sulphate, but determined as CaO by titration with KMnO_4), and a precipitate was obtained containing 77.98 per cent Pb. The building up of the calcium chloride in the subsequent leaches resulted in a poorer grade of precipitate, the lead content falling to 65.54 per cent Pb, when the proportion of CaO in solution was 0.52 per cent. In tests 24 and 25 the addition of lime was omitted, and the lime content of the solution fell to 0.36 per cent, whereas the lead content of the precipitate rose to 70.62 per cent.

In each test an attempt was made to wash out the sodium chloride left in the residue after leaching, the wash water being added to the brine to make up for losses of solution. The volume of solution built up rather fast because too much wash water was generally used, and more sodium chloride had to be added to keep the solution sufficiently saturated. The excess of water was evaporated after cycle 7 and after cycle 30.

In cycles 31 to 35 the object was to determine how much lead could be gotten into solution. In general, an excess of ore was used. In test 31 the lead content in the solution was 0.945 per cent and in test 32 only 0.893 per cent. This low content was probably due to the use of wash water. Hence in test 33 no wash water was added to the solution, and the lead content increased to 1.28 per cent. It was noticed that when calcium chloride solution was added a peculiar white precipitate not resembling calcium sulphate was formed. It was found in leach 35 that if the calcium chloride solution were later saturated with sodium chloride this precipitate did not form. Hence the compound was probably the basic chloride of lead, formed on diluting the brine containing lead with water.

CONCLUSIONS FROM TESTS.

From this series of leaches the following conclusions can be drawn:

1. Cyclic leaching of a roasted lead sulphide ore with a neutral brine, followed by precipitation of the lead from the brine by means of lime, is possible under certain conditions.
2. By removing sulphates from the solution by the use of calcium chloride, followed by careful washing, it is possible to obtain a precipitate of hydrated lead oxide containing 70 to 75 per cent lead.
3. The addition of a small amount of calcium chloride need not be made after every cycle, but only when the content of sulphates seems to be building up to an amount that affects the solubility of the lead in the solution.

TABLE 44.—Results of cyclic leaching of calcines.

Test No.	Assay of solution before precipitation.				Assay of solution after precipitation.				Assay of tailing.				Lead recovery.		CaCl ₂ added.
	Vol. of solution.	Pb.	S.	CaO.	Vol. of solution.	Pb.	S.	CaO.	Weight.	Pb.	S.	CaO.	By solution assay.	By tailing assay.	
	C. c.	Per cent.	Per cent.	Per cent.	C. c.	Per cent.	Per cent.	Per cent.	Grams.	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.	Grams.
1.	1,000	0.56	0.23	0.26	1,000	None.	0.192	0.175	7.60	65.78	0.49	8.66	78	86.5	100.
2.	1,100	0.60			1,200	do.			7.90	62.08	1.74	10.00			100.
3.	1,340	1.13	344	.27	1,420	Trace	.37	.254	9.82	56.69	3.53	16.40	71	87.2	100.
4.	1,430	.53			1,510	do.			9.25	62.68	2.10	13.20			100.
5.	1,500	.113	316	.21	1,510	None.	.315	.36	8.85	71.31	1.10	4.50	79	(?)	100.
6.	1,600	.45				do.			9.60	70.18	.89	6.11			100.
7.	1,800	.36	364	.187	1,800	do.	.30	.192	7.90	69.41	Trace	11.50	87	79	3.02
8.	1,570	.41		Trace		do.			9.15	67.50	1.37				3.07
9.	1,770	.42	292	.155	1,690	do.	1.65	.21	10.10	52.24	3.36	9.12	95	81	3.63
10.	1,690	.39				do.			7.75	71.18	do.				3.92
11.	1,800	.38				do.			8.60	70.28	Trace	3.80			1.20
12.	1,720	.38				do.			7.75	73.44	do.	3.14			1.20
13.	1,720	.40				do.			8.45	70.28	do.	5.80			1.28
14.	1,830	.355				do.			8.10	69.10	1.08	5.23			1.28
15.	1,730	.38	.24	.26		do.	.23		8.50	72.88	.46	3.33	84.5	89	1.28
16.	1,650	.42				do.			8.55	73.66	Trace	2.86			1.28
17.	1,760	.35				do.			8.06	67.80	Trace	4.60			1.28
18.	1,970	.34				do.			8.25	68.36	Trace	2.86			1.28
19.	1,800	.37				do.			8.55	69.18	Trace	2.86			1.28
20.	1,800	.37				do.			8.65	72.54	Trace	3.33			1.28
21.	1,910	.34	.117	.35	2,030	do.	.130	.346	7.75	72.64	Trace	2.14	70	81.5	1.28
22.	1,910	.34		.46		do.			8.15	72.64	Trace	2.14			1.28
23.	1,940	.45		.52		do.			7.35	65.54	Trace	5.10			1.28
24.	1,670	.41	103	.44		do.			9.55	71.18	Trace	5.10			1.28
25.	1,780	.38		.28	1,800	do.	.109	.38	8.75	70.62	Trace	6.90			1.28
26.	1,800	.37		.33	1,900	do.	.33	.33	8.85	75.26	Trace	5.47			1.28
27.	1,830	.40		.30	1,800	do.	.46	.46	8.82	61.12	Trace	6.56			1.28
28.	1,830	.40		.30	1,900	do.	.45	.45	9.35	71.18	Trace	2.81			1.28
29.	1,900	.35		.34	1,910	do.	.30	.30	8.02	71.18	Trace	2.18			1.28
30.	2,040	.28		.10	2,080	do.	.102	.31	8.25	68.93	Trace	6.60			1.28
31.	1,040	.945		.56	1,100	do.	.082	.57	14.65	76.04	Trace	3.32	76	83.9	1.28
32.	1,280	.893		.082	1,500	do.	.068	.57	15.70	76.27	Trace	4.01			1.28
33.	1,200	.128		.30	1,350	do.	.46	.46	17.35	75.14	Trace	4.76			1.28
34.	1,410	.65		.082	1,510	do.	.082	.36	15.60	77.20	Trace	4.04			1.28
35.	620	2.43		.137		do.	.137	.62	20.40	73.55	Trace	5.24			1.28

Assay of calcines.—Pb, 7.78 per cent.; Zn, 3.8 per cent.; CaO, 1.62 per cent.; acid S, 1.65 per cent.; total S, 2.40 per cent.; Ag, 4 ounces per ton.

Average analysis of precipitates.—Pb, 70.83 per cent.; H₂O, 14.50 per cent.; CaO, 4.35 per cent.; S, 0.82 per cent.; Cu, 4.80 per cent.; Mn, 1.43 per cent.; Zn, .66 per cent.; total, 97.11 per cent.

4. The calcium chloride solution added should be saturated with sodium chloride in order to prevent precipitation of a basic chloride of lead by dilution of the brine.

5. The precipitation of lead from the brine by lime takes place quickly both in cold and in warm solutions.

6. The lime should be slaked in brine in order to prevent dilution of the leaching solution.

7. Any sodium chloride carried down with the lead precipitate can be washed out with water.

8. Some chlorine is left in the precipitate, according to the efficiency of washing, and it is probable that this would cause volatilization of lead chloride during smelting of the precipitate.

LEACHING WITHOUT CALCIUM CHLORIDE.

The second series of cyclic leaches was performed on a sample of calcines from a shaft roaster (Holt-Dern) of semicommercial size used at the Bunker Hill & Sullivan test plant. In this series of tests the cycle consisted of leaching with neutral brine followed by precipitation with lime. Calcium chloride was not added to any of the leaches, in order that sodium sulphate might build up in the solution if it would. The numerical data are contained in Table 45.

In cycle 11 the solution was evaporated to a smaller volume after precipitation. During the evaporation a scum of brown material separated but gave a negative assay for lead. Evaporation was necessary because the washing with water diluted the solution and more sodium chloride had to be added to keep the solution saturated. The accumulation of solution was undoubtedly greater than could be allowed in practice and to retain all impurities resort was had to evaporation of the solution to reduce its volume.

In cycle 13 the lead-bearing brine had a yellowish green color, probably due to manganese and a small content of iron. The lead hydrate precipitate was colored brown and the average assay of all the precipitates shows about 1 per cent of manganese.

In cycle 14 large volumes of water were used to wash the sodium chloride out of the precipitate.

In cycle 15, during the analysis for lime, considerable iron and manganese was noted.

In cycle 16 an excess of sodium chloride was added to the saturated brine before precipitation. The lead seemed to be thrown down more rapidly than in previous cycles, precipitation being practically complete in a few minutes.

In cycle 17 an excess of sodium chloride was added to the saturated brine, but the precipitation was difficult and the precipitate held sodium chloride tenaciously.

TABLE 45.—Results of brine leaching of calcine containing 10.7 per cent lead.

Cy- cle.	Time ag- itated.	Solution before precipitation.				Cyclo- needed to pre- cipitate Pb in solution.	Solution after precipitation.				Lead precipitate.				Tailings from leach.							
		Vol- ume.	Lead.	Sulphur.	Lime.		Gms.	P. ct.	Gms.	P. ct.	Gms.	P. ct.	Weight.	Lead con- tent.	Sul- phur.	Lime con- tent.	Weight.	Lead con- tent.	Sul- phur con- tent.	Lime con- tent.	Ex- trac- tion of lead.	C ₂ Cl ₂ sol- ed.
Hours.		Gms.	Gms.	Gms.	Gms.	Gms.	P. ct.	Gms.	P. ct.	Gms.	P. ct.	Gms.	P. ct.	Gms.	P. ct.	Gms.	P. ct.	Gms.	P. ct.	Gms.	P. ct.	Gms.
1	16	1,080	9.50	2.60	0.16	2.50	1,000	2.60	0.21	2.75	0.25	38.6	0.00	9.83	38.6	0.00	10.6	82.5	2.6	0.22	1.3	87.5
2	16	1,650	9.80	2.80	0.17	2.50	1,210	2.30	0.26	3.76	0.35	33.9	0.00	11.1	33.9	0.00	11.1	83.5	2.6	0.22	1.3	92.5
3	16	1,650	9.80	3.00	0.23	2.50	1,230	2.30	0.26	3.76	0.35	33.9	0.00	11.1	16.00	0.00	10.6	83.5	2.6	0.22	1.3	91.5
4	16	1,650	9.80	3.00	0.23	2.50	1,230	2.30	0.26	3.76	0.35	33.9	0.00	11.1	19.70	0.00	6.5	2.0	1.7	1.6	1.00	
5	16	1,650	9.80	3.00	0.23	2.50	1,230	2.30	0.26	3.76	0.35	33.9	0.00	11.1	23.40	0.00	6.0	2.0	1.6	1.48	3.5	82.5
6	16	1,650	9.80	3.00	0.23	2.50	1,230	2.30	0.26	3.76	0.35	33.9	0.00	11.1	17.50	0.00	3.2	2.0	1.3	1.10	3.5	95.5
7	16	1,650	9.80	3.00	0.23	2.50	1,230	2.30	0.26	3.76	0.35	33.9	0.00	11.1	16.25	0.00	3.2	2.0	1.3	1.00	3.2	96.5
8	16	1,650	9.80	3.00	0.23	2.50	1,230	2.30	0.26	3.76	0.35	33.9	0.00	11.1	18.35	0.00	3.8	2.0	1.3	0.79	3.9	89.5
9	16	1,650	9.80	3.00	0.23	2.50	1,230	2.30	0.26	3.76	0.35	33.9	0.00	11.1	15.60	0.00	3.5	2.0	1.1	1.14	2.0	89.5
10	16	1,650	9.80	3.00	0.23	2.50	1,230	2.30	0.26	3.76	0.35	33.9	0.00	11.1	17.15	0.00	7.0	2.0	1.1	1.1	3.5	70.0
11	16	1,650	9.80	3.00	0.23	2.50	1,230	2.30	0.26	3.76	0.35	33.9	0.00	11.1	18.25	0.00	11.0	2.0	0.9	1.26	3.5	88.0
12	16	1,650	9.80	3.00	0.23	2.50	1,230	2.30	0.26	3.76	0.35	33.9	0.00	11.1	14.00	0.00	4.0	2.0	0.71	1.71	2.7	76.0
13	16	1,650	9.80	3.00	0.23	2.50	1,230	2.30	0.26	3.76	0.35	33.9	0.00	11.1	12.31	0.00	4.1	2.0	1.0	1.0	2.7	76.0
14	16	1,650	9.80	3.00	0.23	2.50	1,230	2.30	0.26	3.76	0.35	33.9	0.00	11.1	13.30	0.00	6.8	2.0	2.0	1.0	2.5	72.0
15	16	1,650	9.80	3.00	0.23	2.50	1,230	2.30	0.26	3.76	0.35	33.9	0.00	11.1	13.30	0.00	6.8	2.0	1.0	1.0	2.5	72.0
16	16	1,650	9.80	3.00	0.23	2.50	1,230	2.30	0.26	3.76	0.35	33.9	0.00	11.1	13.30	0.00	6.8	2.0	1.0	1.0	2.5	72.0
17	16	1,650	9.80	3.00	0.23	2.50	1,230	2.30	0.26	3.76	0.35	33.9	0.00	11.1	13.30	0.00	6.8	2.0	1.0	1.0	2.5	72.0
18	16	1,650	9.80	3.00	0.23	2.50	1,230	2.30	0.26	3.76	0.35	33.9	0.00	11.1	13.30	0.00	6.8	2.0	1.0	1.0	2.5	72.0
19	16	1,650	9.80	3.00	0.23	2.50	1,230	2.30	0.26	3.76	0.35	33.9	0.00	11.1	13.30	0.00	6.8	2.0	1.0	1.0	2.5	72.0
20	16	1,650	9.80	3.00	0.23	2.50	1,230	2.30	0.26	3.76	0.35	33.9	0.00	11.1	13.30	0.00	6.8	2.0	1.0	1.0	2.5	72.0
21	16	1,650	9.80	3.00	0.23	2.50	1,230	2.30	0.26	3.76	0.35	33.9	0.00	11.1	13.30	0.00	6.8	2.0	1.0	1.0	2.5	72.0
22	16	1,650	9.80	3.00	0.23	2.50	1,230	2.30	0.26	3.76	0.35	33.9	0.00	11.1	13.30	0.00	6.8	2.0	1.0	1.0	2.5	72.0
23	16	1,650	9.80	3.00	0.23	2.50	1,230	2.30	0.26	3.76	0.35	33.9	0.00	11.1	13.30	0.00	6.8	2.0	1.0	1.0	2.5	72.0
24	16	1,650	9.80	3.00	0.23	2.50	1,230	2.30	0.26	3.76	0.35	33.9	0.00	11.1	13.30	0.00	6.8	2.0	1.0	1.0	2.5	72.0
25	16	1,650	9.80	3.00	0.23	2.50	1,230	2.30	0.26	3.76	0.35	33.9	0.00	11.1	13.30	0.00	6.8	2.0	1.0	1.0	2.5	72.0
26	16	1,650	9.80	3.00	0.23	2.50	1,230	2.30	0.26	3.76	0.35	33.9	0.00	11.1	13.30	0.00	6.8	2.0	1.0	1.0	2.5	72.0
27	16	1,650	9.80	3.00	0.23	2.50	1,230	2.30	0.26	3.76	0.35	33.9	0.00	11.1	13.30	0.00	6.8	2.0	1.0	1.0	2.5	72.0
28	16	1,650	9.80	3.00	0.23	2.50	1,230	2.30	0.26	3.76	0.35	33.9	0.00	11.1	13.30	0.00	6.8	2.0	1.0	1.0	2.5	72.0
29	16	1,650	9.80	3.00	0.23	2.50	1,230	2.30	0.26	3.76	0.35	33.9	0.00	11.1	13.30	0.00	6.8	2.0	1.0	1.0	2.5	72.0
30	16	1,650	9.80	3.00	0.23	2.50	1,230	2.30	0.26	3.76	0.35	33.9	0.00	11.1	13.30	0.00	6.8	2.0	1.0	1.0	2.5	72.0

In cycle 21 an excess of sodium chloride was added to the brine before leaching, and the solution was evaporated after precipitation. A black scum separated during evaporation, but it contained no lead. The salt crystallizing out during evaporation contained no lead and a considerable amount of lime and of sulphate sulphur.

In cycle 23 an excess of sodium chloride was used and, after precipitation, the lime was added slowly, resulting in a high-grade precipitate.

In cycle 27 enough calcium chloride was added to precipitate the sulphur in order to determine whether it would improve the leaching. The higher extraction in cycle 28 seems to be the result of this. However, a lower grade of precipitate in cycle 27 was caused.

SUMMARY OF RESULTS OF TESTS.

The results of the tests shown in Table 45 may be summarized as follows:

1. Sulphates did not build up in the solution to a content of more than 0.6 per cent sulphur. With higher contents calcium sulphate and sodium sulphate often separated as a scum or stayed behind in the tailing. Washing with water to recover sodium chloride from the tailing would remove the sodium sulphate, which was only slightly soluble in the saturated brine.

2. Sulphates were not precipitated with the lead hydrate in sufficient quantity to appreciably lower the lead tenor of the precipitate.

3. Rapid and complete precipitation was found difficult whenever powdered lime was used. Milk of lime is recommended, and preferably it should be made up in a saturated brine solution.

4. Precipitation was found to take place as readily in cold as in hot solutions.

5. Tests with phenolphthalein indicated that when even an excess of lime was used for precipitation the saturated brine would dissolve only a small proportion of the excess lime. Hence an excess of lime merely dilutes the precipitate and does not injure the leaching solution.

6. The lead precipitate is readily freed of sodium chloride by washing with cold water.

7. A lead hydrate precipitate containing 70 to 75 per cent lead can be obtained by careful work.

8. Excessive fouling of the solution with sulphates did not occur. The solution is known to be capable of holding 12 to 15 grams of lead per liter, but was so proportioned that not more than 10 grams per liter were used, in order that all the lead in the ore might be dissolved.

LATER LEACHING TESTS AT BUNKER HILL & SULLIVAN PLANT.

The results of these two series of cyclic leaches indicated that trouble from fouling of the solution was not liable to be serious. However, shortly afterward the experimental plant at the Bunker Hill & Sullivan mill was put into good condition and all leaks were stopped in order that the cyclic leaching might be tested on a larger scale. The solutions immediately began to foul, and finally reached a stage where they would hardly dissolve any lead from the ore. Then the proposal to introduce calcium chloride for the removal of sulphates was adopted. The lead-dissolving power of the solutions immediately improved.

DESCRIPTION OF TESTS.

The plant was shut down to permit studying this problem. The effects of increasing the proportion of calcium chloride were tested in various leaches, with the result that whenever the calcium chloride in the brine was increased the extraction of lead fell off slightly, whereas the silver extraction suffered severely. Hence it does not seem advisable to use any more calcium chloride than is necessary to precipitate the sulphate sulphur in the solution.

Washing the calcines with water before leaching with brine, to determine whether any of the fouling elements, such as sulphates, could be removed in this way, showed that 38.4 per cent of the sulphate sulphur in the ore was soluble and 77.1 per cent of the manganese. By the use of calcium chloride, to prevent fouling, 60 per cent of the total sulphates in the ore were converted into insoluble calcium sulphate, and at the same time 28.56 per cent of the manganese was prevented from appearing in the brine leaches. By prewashing with water and using calcium chloride in the brine 84 per cent of the sulphates and 98 per cent of the manganese failed to appear in the brine leach.

To confirm these results larger quantities of calcines were treated and the conclusions are quoted from the report of C. L. Larson, who was then in charge of the experimental work at the Bunker Hill & Sullivan plant.

1. The use of a water wash preliminary to brine leaching decreases the sulphate fouling 52 per cent and the manganese fouling 77 per cent.
2. Zinc does not appear to build up in the solution to any great extent, owing to its complete precipitation with the lead by lime.
3. The belief that lead sulphate reacts with the sodium chloride in the brine to form lead chloride and sodium sulphate is further supported by the observation in these series of tests that the first effluent from a leaching tank contains more sulphur than that corresponding to the lead in solution, whereas the subsequent runnings contain lead in excess of the amount equivalent to the sulphate sulphur.

With these points in mind, a series of cyclic leaches of blast-waster calcine was made with neutral brine by Larson in the company's laboratory. Forty liters of saturated brine was prepared as a stock solution and about 10 pounds of ore per charge was leached in earthenware jars. The brine solutions were precipitated with lime and filtered. Any alkalinity of the filtrate was neutralized with hydrochloric acid. In the first leach about 4 tons of solution per ton of ore were used, followed by one or two more brine leaches, and finally a water wash to recover sodium chloride from the tailing. The wash water from the tailing and from the lead hydrate precipitate was evaporated to saturation and added to the stock solution. In spite of this precaution the amount of solution decreased markedly in the 18 cycles, and might well have been replaced by new brine if the specific purpose of these tests had not been to determine the effect of fouling when no new brine could be introduced.

The first nine cycles were on material that had received a preliminary washing with water and did not deteriorate very fast. After that unwashed material was used to make the fouling agents build up faster. At the completion of cycle 17 the solution had so deteriorated that only about 8 grams of lead per liter could be dissolved, hence the solution was treated with calcium chloride. The chlorine content before treatment with calcium chloride had fallen to 165 grams per liter. After precipitation it rose to 182 grams per liter and the solubility of lead in the solution increased in about the same proportion, as was evidenced by the first runnings of the next leach.

CONCLUSIONS FROM RESULTS OF TESTS.

Larson's conclusions on the cyclic operations, are as follows:

1. Neutral brine, used cyclically, extracts about 99 per cent of the soluble lead formed in blast roasting (which is 85 to 99 per cent of the lead in the ore) within certain limits of fouling. In other words, with fouling taken care of, the lead extraction of the neutral brine process depends entirely on the sulphating efficiency of the roasting step of the process, and it has been shown that this is 85 to 99 per cent.

2. Neutral brine gives erratic results with silver. In general the only conclusion that can be definitely drawn is that the better the sulphating of the lead the better is the extraction of the silver. Acidified brines are more reliable. A high content of calcium chloride in the solution causes lower extractions of silver.

3. Considerable manganese was soluble in the brine but did not build up, owing to complete precipitation by the lime. The manganese does, however, lower the grade of the precipitate markedly.

4. Sulphur builds up steadily with the continued use of the brine. No maximum of sulphate sulphur seemed to be reached, the highest concentration, in cycle 17, being 11.08 grams per liter. Long before this amount was reached it was seen that the solution was deteriorating in its ability for dissolving lead. However, the sulphates need only be eliminated intermittently and can be eliminated very well by the use of crushed solid calcium chloride. One pound of precipitated calcium sulphate is obtained by the addition of every pound of calcium chloride. It is possible that some of this could be sold for gypsum.

5. The use of a preliminary water wash greatly prolongs the efficient life of the lixiviant and removes manganese from the system, resulting in a higher grade of precipitate.

6. Practically complete precipitation of lead is obtained by the use of lime. Manganese and zinc are also completely precipitated. The silver, however, is not completely precipitated and the brines usually carried 4 to 6 milligrams of silver per liter after lime precipitation.

7. The precipitate, if not washed, contained 55 to 60 per cent lead and after washing with water, 65 to 70 per cent lead.

8. Washing the precipitate was difficult, owing to the tendency of the filter cake to crack quickly. Repulping of precipitate with fresh water, followed by filtration, was found necessary in order to remove the sodium chloride from the cake. With the larger amounts of precipitate handled in these tests, it was difficult to obtain a cake containing less than 5 per cent of chlorine. The rest of the chlorine seems to be locked up in a complex lime-lead chloride. It was more difficult to bring the lime and solution into efficient contact in these tests and in the test plant, and this may account for the higher amount of insoluble chlorine in the precipitate as compared with the work performed at the Salt Lake City station.

9. Washing the tailing with water and evaporating the washings in order to recover sodium chloride from the tailing is feasible, if the salt is worth the trouble. Such washing tends, however, to return to the system some of the sodium sulphate left in the tailing.

10. No advantage is apparent in conducting the operations at temperatures other than the ordinary mill temperature. Heating of solution in a mill is generally by introduction of exhaust steam into the solution, which is undesirable in this process because the solution should always be saturated with sodium chloride. In this connection it was noted that in test 12, when the temperature dropped very low over night, sodium sulphate crystallized out of the solution.

Larson's conclusions on fouling meet the authors' approval although they doubt the advisability of using a preliminary water

wash to remove soluble fouling agents. The water left in the pulp will tend to dilute the brine later applied, also as much moisture must leave the leaching tanks as was in the water-washed pulp before leaching. These conditions do not permit partial recovery of salt by washing with water after leaching with brine. Where the brine is applied to dry ore without washing with water, it will be necessary to add water at the end of the cycle to keep up the volume of mill solution. As it has been definitely shown that the use of calcium chloride is sufficient to prevent fouling, and that the chlorine content of the solution must be kept up near the point corresponding to saturation with sodium chloride, these are the two conditions that would have to be watched in large-scale work. The company is now testing out this and other improved processes. It is to be hoped that the results of the work will be published at an early date.

Since the time of writing this bulletin some of the results have been described by Larson^a in an article published in the Mining and Scientific Press.

As acidified brine must be used in order to get satisfactory extractions of the silver, and sulphuric acid would probably be the acid used, on account of its cheapness, the only disadvantage would be the more frequent additions of calcium chloride necessary in order to keep the resulting sodium sulphate from building up too fast.

There is considerable difficulty in getting good extractions of silver from this type of ore. Later experiments have led the writers to think that much of the difficulty is due to the reprecipitation of silver from the solution on unroasted zinc sulphide. The lead is often roasted completely to the sulphate or oxide form before the zinc sulphide is much affected, and when working on complex sulphides it has been found that the unroasted sphalerite could precipitate large amounts of silver from brine solutions. Consequently the longer the time of contact of the leaching solution with an ore containing a small amount of zinc sulphide, the more silver will be precipitated and the lower the extraction. On that account a roaster that will efficiently roast the sphalerite, as well as the galena, is desirable. Amalgamation, cyanidation, hyposulphite lixiviation, leaching with acid brine, brine containing cupric chloride, brine containing ferric chloride, and other methods were tried in an effort to extract silver from badly roasted ore. In all tests it was found difficult to get extractions that would even parallel those obtained with acid brine leaches. By roasting the ore properly and leaching with acid brine it was found that as much as 80 per cent of the silver could be extracted, and that usually the lead was practically all converted to the sulphate in these good roasts.

^a Larson, C. L., Hydrometallurgy of lead-silver at the Bunker Hill smelter: Min. and Sci. Press, Aug. 25, 1917, pp. 275-279.

LEACHING UNROASTED GALENA.

Galena is readily converted to the sulphate by roasting, and great care does not have to be taken to obtain practically all of the lead in the form of lead sulphate. Seemingly lead sulphide is the most easily sulphated metal sulphide ordinarily met with.

Some leaching tests with acidified brine of unroasted galena showed that more than one-third of almost any galena sample tested would dissolve in acidified brine in two hours. Hydrogen sulphide was generated by the reaction. Sphalerite was practically unaffected under the same conditions. Even if the roasting is improperly done, the lead, so far as that metal is concerned, is liable to be removed successfully in spite of being incompletely oxidized or chloridized.

PRECIPITATION TESTS.

Precipitation of the lead in solutions from the leaching of roasted sulphide ores of lead differs in no respect from the precipitation of lead in the solutions from the leaching of oxidized ores. Either precipitation with lime, resulting in a basic lead hydrate, or electrical precipitation, yielding metallic lead, may be used. The latter method would be preferable where large amounts of soluble impurities, like zinc sulphate and manganese sulphate, tended to collect in the solution, as such impurities might contaminate the lead hydrate precipitate obtained by the use of lime.

Some tests by Larson of the efficiency of lime in precipitating lead from brine solutions gave rather unexpected results. It was found that lime could be used in proportions considerably less than those theoretically equivalent to the lead in solution, with practically complete precipitation. The lead is completely precipitated with as little as 75 per cent of the theoretical equivalent of lime and 96 per cent is precipitated with 65 per cent of the theoretical amount of lime. The precipitates were washed with hot water on a filter. Analysis showed considerable lime and chlorine in the precipitate. Because of the contamination of the precipitate with zinc, manganese, calcium sulphate, and other impurities, it was difficult to calculate the composition of the lead compound formed in precipitation. However, the uniformly high calcium and chlorine content of the precipitate lead the writers to believe that a basic chlorine of lead, or a basic double chloride or lead and calcium, is formed.

CONCLUSIONS FROM RESULTS OF TESTS.

From the tests that have been described the following conclusions can be drawn:

1. Practically all of the zinc in the solution is precipitated with the lead. With the material tested the precipitate contained about 1 per cent zinc.

2. Only about one-half of the manganese was precipitated in the tests, in which less than the theoretical proportion of lime to lead was used.

3. Less than 70 per cent of the proportion of lime theoretically required can be used, with complete precipitation of the lead.

4. Silver is not completely precipitated by lime and the smaller the amount of lime used relatively to the lead the less complete will be the precipitation of the silver from the solution.

5. The greater the deficiency of lime below the theoretical requirements the higher will be the grade of the "lead hydrate" precipitate.

6. The CaO balance sheets (not given herein) for the tests show that some calcium chloride was produced during precipitation. About one-half to one-third of the calcium applied in the form of hydrate reappeared in the neutral precipitated solution as chloride and sulphate.

Purification of the solution, to improve the grade of lead precipitate formed, was not found necessary in most of the tests, except for the addition of calcium chloride to precipitate the sulphates. The use of ground limestone to remove iron, alumina, and other substances was not tested on solutions from sulphide ores, as that feature was not developed until after the tests were finished. Operation of the Bunker Hill & Sullivan semicommercial plant revealed that it was difficult to wash the lead precipitate efficiently in large-scale practice. For that reason the precipitation of lead by electrolysis was tested in a tank capable of holding 5 tons of solution. The lead sponge obtained was so easily melted into lead bullion that no further attempt to purify the solutions was made. It is believed, however, that the use of crushed limestone would go far to mitigate the difficulty of making a high-grade lead-hydrate precipitate in a commercial plant, in the same way that it improved conditions in the small scale tests at the Salt Lake City station.

Silver precipitates so well on scrap iron that precipitation of the silver on scrap iron, followed by electrolytic precipitation of the lead is preferable, because the purity of the lead-sponge precipitate is little affected by the iron in solution. This method allows the preparation of desilvered lead direct.

CONCLUSIONS FROM TESTS OF SIMPLE SULPHIDE ORES.

In summing up the results of the tests of simple sulphide ores of lead, the following may be said:

1. If simple sulphide ores of lead are given a chloridizing roast at temperatures of not more than 100° C., in either a reverberatory roasting furnace or in blast roasters of any type, practically all of the lead sulphide is converted into lead sulphate and part of the silver associated with the lead is chloridized.

2. The sulphated-lead compounds and the chloridized silver can be dissolved from the roasted material by leaching with an acidified saturated solution of sodium chloride. Acidification of the brine is necessary in order to convert any oxide of lead to a soluble form; also much better extractions of silver result.

3. The lead and the silver may be precipitated as metals by electrolysis, or as hydrates (or other compounds) by the addition of lime. If lime is used, a precipitate containing 60 to 80 per cent of lead, depending on the impurities present and the conditions of precipitation, is formed.

4. The brine, after precipitation of the lead and the silver, can be used for further leaching.

5. The costs of the proposed process have already been discussed under the heading of "Oxidized ores" (p. 109). It seems probable that in treating simple sulphide ores by this process the costs will approximate about \$2.50 per ton of ore. This process should be a serious competitor of concentration followed by shipment of concentrate to a lead smelter, because the recovery of metal is high, and metallic desilverized lead can be made, which lessens transportation costs.

COMPLEX SULPHIDES CONTAINING LEAD.^a

Various combinations of sulphides of metals have come to be designated as "complex sulphides." Ordinarily this term is applied to mixtures of zinc and lead sulphides. It is also applied to mixtures of zinc, lead, and iron sulphides. In some localities are found mixtures of zinc, lead, iron, and copper sulphides, often accompanied by gold and silver, and these are also called complex. Mixtures of galena and pyrite are not often called complex, because in lead smelting the iron enters the slag and causes little trouble beyond lowering the lead assay of the ore.

As mentioned later in this report, the metallurgical treatment of the latter ores depends upon the separation of the lead, iron, and copper from the zinc minerals, as zinc is a source of loss in both lead and copper smelters. Lead, iron, and copper minerals in admixture can be smelted in the lead blast furnace by leaving enough sulphur in the roasted ore to form some leady copper matte. The gold and the silver distribute themselves between the matte and the metallic lead formed in the furnace, and any zinc goes either into the slag or into the flue dust. Lead in a zinc ore is usually penalized and is not only often left in the residues thrown out from the zinc smelter, but the spelter from such an ore is of lower grade. Rather heavy charges are exacted by the lead smelters for treating the residue from zinc smelters.

^a Experimenter, Marvin J. Udy.

LEADY ZINC ORES AND CONCENTRATES.

To the treatment of zinc ores or concentrates now being sent to the smelters and which still carry considerable amounts of lead, chloridizing blast roasting followed by brine leaching was deemed especially applicable. Table 46 shows the analyses of a number of complex sulphide products, mostly zinc concentrates containing lead not ordinarily capable of removal by ordinary ore-dressing methods. In fact, most of these products have resulted from the best ore-dressing methods that can be economically applied to such ores. For comparison the average of 3,800 lots of ore from the Joplin zinc mills is given in an analysis by Waring.^a The Joplin zinc ores are "free" milling and most of the lead can be recovered by gravity methods of ore dressing, although occasional lots of Joplin zinc ore carry percentages of lead much higher than 0.7 per cent, the content shown in the average analysis given. In the other zinc concentrates from various localities the lead content varied from 1.53 to 12.85 per cent. If the shipper of the ore wishes to send the residue to a lead smelter after the zinc has been removed in the zinc smelter, the lead smelter will pay for only 60 per cent of the lead and silver in the material and at prices considerably below the usual market price. The losses, freight, and smelting charges on such material average \$8 to \$10 per ton and consequently it rarely pays to resmelt leady zinc ore. Usually such an ore will be sent to the pigment plants, which make a mixed zinc-lead pigment and buy the ore on the combined zinc and lead content. It is a common practice to see zinc concentrate or ore containing 5 to 8 per cent lead sent to the zinc smelter for its zinc content alone.

^a Waring, G. W., Zinc ores of the Joplin district, their composition, character, and variation: Bull. Am. Inst. Min. Eng., Sept., 1917, pp. 1257-1266.

TABLE 46.—*Results of analyses of zinc concentrates.*

[Analysts, M. J. Udy and E. B. Barrett.]

Material treated.	Source.	Insoluble.	CaO.	Fe.	Al ₂ O ₃ .	MgO.	Mn.	As.	S.	Zn.	Pb.	Cu.	Ag.	Au.
Daly Judge, concentrates.	Park City, Utah.	Per ct. 4.1	Per ct. 4.95	Per ct. 6.1	Per ct. 5.22	Per ct. Tr.	Per ct. 2.0	Per ct. 0.52	Per ct. 30.4	Per ct. 43.73	Per ct. 3.83	Per ct. 1.38	Oz. per ton. 18.45	Oz. per ton. 0.015
Timber Butte, flotation concentrate.	Butte, Mont.	3.6	1.75	4.4	3.6			.55	33.1	53.9	1.33	2.52	16.0	.04
Do.	do.	3.6	1.88	8.6	8.6			.40	23.8	48.6	1.85	1.85	10.8	.03
Frisco, machine concentrate.	Wallace, Idaho.	8.48	1.79	13.5	5.9				19.3	38.8	10.0	None.	12.74	None.
Do.	do.	1.96	1.89	13.0	2.0				26.3	39.6	10.4	None.		None.
Highland Surprise, fine zinc concentrate.	Pine Creek, Idaho.	4.92	1.65	12.1	6.0				22.0	43.8	6.7	None.	3.2	
Success, flotation zinc concentrates.	Habana, Cuba.	16.72	.99	11.5	2.3				31.1	37.5	None.	8.17	12.84	.08
Marsh, flotation zinc from tables.	Wallace, Idaho.	14.8	1.04	7.7	10.8				23.8	37.2	12.85	None.	1.38	None.
Comet, zinc ore.	do.	5.4	Tr.	12.7	11.8				25.2	37.4	10.7	None.	3.24	None.
Hercules, flotation zinc concentrates.	Wallace, Idaho.	5.92		25.12	12.0				33.9	21.1	7.72	1.49	18.36	24
3,800 lots ^a .	Joplin, Mo.	4.8	1.17	11.3	11.17				26.9	44.9	9.69	2.31	7.10	None.
Mary Murphy, flotation concentrates.	Romley, Colo.	14.6	1.05	2.23		1.7	.01		30.7	58.3	.70	.05	10.25	15
Mary Murphy, feed to electric machines.	do.	7.5		5.45			2.25		22.1	39.5	11.7	2.12	5.1	.42

^a Analysis by G. W. Waring.

TESTS OF ZINC CONCENTRATES.

In applying the method of chloridizing roasting followed by brine leaching, several types of zinc concentrate were tested. One was finely divided flotation concentrate and the other was coarser table concentrate, in order that the effect of size of particles on the roasting might be observed. For better comparison flotation concentrate and table concentrate from the same mill (Mary Murphy mine, Romley, Colo.) were used. Their analyses were as follows:

Results of analysis of table concentrate and flotation concentrate.

	Mixed flotation concentrate.	Electro- static feed.
Insoluble.....per cent..	14.6	7.50
Iron.....do.....	2.82	11.0
Zinc.....do.....	39.5	31.28
Lead.....do.....	11.7	11.6
Copper.....do.....	2.12	1.14
Silver.....ounces per ton..	10.25	(a)
Gold.....do.....	.15	(a)
Sulphur.....per cent..	22.35	29.75

a Undetermined.

These materials were roasted on the down-draft roaster invented by Christensen, and described on page 68. It is fully the equivalent of a Dwight-Lloyd sintering machine in its action and the same operation could be performed in such a machine. Mixtures containing various amounts of salt, moisture, fuel, etc., were tested. The suction was high on some and low on others. Usually the ore would burn, after ignition, with no addition of fuel, but in some tests powdered coal was added to give higher roasting temperatures. The increase of the draft on the ore also gave higher temperatures. Usually the object was to run the roast at such a temperature that the material would not be sintered, although a number of tests to determine the effect of temperatures above the sintering point were attempted. The change in weight in the charge was observed. The weight of lead in the calcine subtracted from the weight of lead in the heading gave the amount of lead volatilized as chloride, sulphate, etc. A 100-gram sample of the calcine was then leached with neutral brine and another 100 grams with acidified brine in order to compare the effects of neutral and acid solutions in removing lead sulphate or chloride and in converting oxidized lead compounds to one of these forms. Any silver chloride formed in roasting would also supposedly be leached, together with some copper.

The mixed flotation concentrate was used for the tests of flotation material in order to determine what could be done in treatment of such a mixture, which is usually tabled in order to form a lead con-

centrate and a zinc concentrate, with more or less imperfect separation. Likewise, the electrostatic feed consisted of the mixed zinc-iron concentrates from the Willey tables of the mill, and invariably contained considerable lead sulphide as well. These conditions are similar to those in practically all mills treating mixed sulphide ores. Electrostatic separation is more or less costly and never perfect. Hence it was thought best to attempt to recover the lead from the electrostatic feed instead of from the zinc concentrate made in the electrostatic machine. However, two roasts of electrostatic zinc concentrate were made in order to see how much lead could be removed by the chloridizing method after the electrostatic concentrator had done the best work possible for it to do.

RESULTS OF TESTS.

The data on the roasting and leaching are contained in Tables 47 to 58.

Tables 47 and 53, containing the roasting data, give the weight of material used in the roast and the weight of the resulting calcine. The proportions of salt, water, and coal added to the material in making up the roasting charge are expressed as percentages of the weight of material to which they were added. The suction applied to the charge on the down-draft roaster is expressed in inches of water-gage.

Tables 48 and 54 contain the data from which was calculated the amount of each metal that volatilized. From the analyses of the headings and calcines and from their respective weights it is possible to calculate the weight of each metal in these products and from the loss to calculate the weight of metal volatilized. The volatilization is expressed in percentage of the total metal content.

Tables 49 and 55 contain the leaching data on the lead in the material for each test. On each calcine two leaches were made, one with neutral saturated brine and the other with acidified saturated brine. The first column in the table shows the number of the roast and the second column the number of the leach. The odd-numbered leaches were with acidified brine and the even-numbered leaches with neutral brine. The weight of calcine used in leaching is shown in the third column, and the percentage of lead in the fourth. The fifth shows the percentage of acid in the leaching solution, the sixth, the volume of solution, and the seventh, the percentage of lead in the leaching solution after leaching. The eighth column shows the number of grams of lead leached out of the material. From these data are calculated the percentage extraction of the lead from the calcines, shown in the ninth column, and also the percentage of the total lead leached referred to the original ore, shown in the tenth column. Column 11

shows the weight of the tailing, column 12 the percentage of lead, and column 13 the weight of lead contained in the tailing. From these latter analyses it is possible to again calculate the percentage of extraction of lead referred to the calcine leached, and also referred to the original material, as shown in columns 14 and 15. The extractions calculated from the analyses of the solutions should agree with the extractions calculated from the analyses of the tailings and should furnish a check on the work. In practice, however, it is found that measurements on the solutions are less accurate than those on the tailing and the percentage of extraction calculated from the tailing analyses are the ones accepted as most reliable.

The zinc tables (Nos. 50 and 56) and the copper tables (Nos. 51 and 57) are calculated in the same way as were the lead tables.

Tables 52 and 58, containing the extraction summary, show the number of the roast, the number of the leach following the roast, and whether the leach was acid or neutral. The percentage of the total lead volatilized, the percentage of the total lead leached, and the sum of these two or the percentage extraction are shown. Similar data for zinc and copper are given. The lead, zinc, and copper tables contain the analyses and the weights of the various products, hence these data need not appear in the summary.

TABLE 47.—*Data on roasting for tests of mixed flotation concentrate.*

Roast No.	Weight of material taken.	Weight of calcine.	NaCl used.	Water used.	Coal.	Draft suction, inches of water.
	Ounces.	Ounces.	Per cent.	Per cent.	Per cent.	
1	48	46	6	10.0		2.0
2	48	47	10	10.0		2.0
3	43	48	6	10.0		.25
4	48	50	10	10.0		.5
5	48	48	6	10.0	2	1.0
6	48	48	10	10.0	5	2.5
7	48	43	6	11.6		.5
8	48	46	10	10.0		2.0
9	48	45	10	9.7	2	2.5
10	48	51	20	10.0	2	7.5
11	48	47	7	10.0		3.0
12	48	48	7	10.0		.5
13	48	51		8.9	(a)	1.0

a 10 per cent pyrite added.

NOTE.—Roasts 11, 12, and 13 were mixed in a laboratory ball mill with saturated brine, drained, and dried to about 10 per cent moisture content.

TABLE 48.—*Volatilization data for tests of mixed flotation concentrate.*

[Assay of heading: Pb, 11.70 per cent; Zn, 39.50 per cent; S, 27.60 per cent; Fe, 2.82 per cent; Ag, 10.20 ounces per ton; Au, 0.20 ounce per ton.]

Roast No.	Lead.			Zinc.			Copper.		
	Content in heading.		Percent- age Volat- ilized.	Content in calcine.		Percent- age Volat- ilized.	Content in calcine.		Percent- age Volat- ilized.
	Per cent.	Ounces.		Per cent.	Ounces.		Per cent.	Ounces.	
1.	11.70	5.61	36.00	39.50	18.98	17.91	1.37	0.630	15.65
2.	11.70	5.61	62.50	39.50	18.98	18.30	1.48	.955	17.95
3.	11.70	5.61	21.20	39.50	18.98	18.20	1.37	.660	11.50
4.	11.70	5.61	38.50	39.50	18.98	19.00	1.37	.985	8.50
5.	11.70	5.61	51.70	39.50	18.98	19.05	1.33	.765	14.1
6.	11.70	5.61	18.90	39.50	18.98	18.85	1.32	.790	12.48
7.	11.70	5.61	33.70	39.50	18.98	40.20	1.61	.692	17.45
8.	11.70	5.61	76.50	39.50	18.98	19.00	1.36	.745	19.95
9.	11.70	5.61	32.30	39.50	18.98	18.30	1.36	.745	19.26
10.	11.70	5.61	34.80	39.50	18.98	18.30	1.42	.974	40.43
11.	11.70	5.61	71.50	39.50	18.98	19.95	.96	.446	19.75
12.	11.70	5.61	92.00	39.50	18.98	40.20	1.14	.715	2.2
13.	11.70	5.61	71.50	39.50	18.98	18.60	1.36	.341	25.60

TABLE 49.—Data on lead extraction in tests of mixed flotation concentrate.

Roast No.	Leach No.	Calcine.		Solution.				Tailing.				
		Weight, grams.	Lead content, per cent.	Sulphuric acid content, per cent.	Volume, c. c.	Lead content.		Weight, grams.	Lead content.		Percentage of ex- traction.	
						Per cent.	Grams.		Per cent.	Grams.	From calcine.	From original material.
1.	1.	100	7.81	0.5	570	0.420	2.39	88	5.75	5.05	35.30	22.70
2.	2.	100	7.81		625	.201	1.25	88	7.70	6.75	13.60	8.70
2.	3.	100	4.47	0.5	580	.270	1.56	83	4.02	3.34	25.30	9.45
2.	4.	100	4.47		580	.132	.76	89	4.02	3.68	17.65	6.57
3.	5.	100	9.20	0.5	590	.302	2.13	82	8.41	6.90	25.00	19.70
3.	6.	100	9.20		590	.253	1.49	88	8.41	7.40	19.30	15.20
4.	7.	100	6.90	0.5	710	.400	3.26	83	4.55	3.78	45.25	27.80
4.	8.	100	6.90		700	.373	2.60	87	4.36	3.80	43.50	26.79
5.	9.	100	5.65	0.5	610	.391	2.38	80	4.40	3.52	37.70	18.20
5.	10.	100	5.65		610	.333	2.03	87	4.80	4.17	26.30	12.70
6.	11.	100	9.50	0.5	670	.402	2.69	80	8.67	6.94	27.00	21.90
6.	12.	100	9.50		600	.373	2.23	84	9.30	7.80	19.35	15.70
7.	13.	100	8.67	0.5	800	.545	4.35	83	4.82	4.00	53.90	35.70
7.	14.	100	8.67		550	.126	.68	89	8.76	7.82	8.80	5.82
8.	15.	100	2.89	0.5	480	.224	1.07	80	2.48	1.98	31.50	7.45
8.	16.	100	2.89		510	.051	.26	83	2.35	1.85	8.31	2.85
9.	17.	100	9.56	0.5	550	.402	2.22	80	9.62	7.70	19.50	15.30
9.	18.	100	9.56		530	.126	.67	89	9.95	8.85	7.41	5.81
10.	19.	100	7.20	0.5	490	.247	1.21	81	6.79	5.50	23.60	15.40
10.	20.	100	7.20		780	.161	1.25	79	6.91	5.45	24.20	15.80
11.	21.	100	3.06	0.5	550	.211	1.32	81	2.28	1.84	39.90	10.25
11.	22.	100	3.06		570	.224	1.27	82	2.38	1.95	36.30	8.00
12.	23.	100	7.97	0.5	570	.202	1.15	84	1.03	.95	51.70	8.90
12.	24.	100	7.97		630	.159	1.00	87	1.03	.96	50.71	8.70
13.	25.	100	2.80	0.5	620	.184	1.14	85	1.89	1.60	43.00	11.05
13.	26.	100	2.80		610	.086	.49	88	2.35	2.06	28.30	9.80

TABLE 50.—Data on zinc extraction in tests of mixed flotation concentrate.

Roast No.	Leach No.	Calclne.		Solution.			Tailing.					
		Weight, grams.	Zinc content, per cent.	H ₂ SO ₄ content, per cent.	Volume, c. c.		Weight, grams.	Zinc content.		Percentage of ex- traction.		
								Per cent.	Grams.		Per cent.	Grams.
1	1	100	39.00	0.5	570	0.575	3.28	88	40.00	35.20	9.75	9.55
1	2	100	39.00	0.5	625	.325	2.03	88	42.00	36.60	5.30	5.20
1	3	100	39.00	0.5	580	.300	2.90	83	42.00	35.30	9.10	9.17
2	2	100	39.00	0.5	580	.200	4.12	89	43.00	38.20	9.05	9.05
4	5	100	38.00	0.5	590	.700	1.16	86	41.30	34.00	12.5	10.08
3	3	100	38.00	0.5	590	.400	2.36	88	41.30	34.00	17.07	17.07
3	6	100	38.00	0.5	590	.800	3.07	88	40.00	32.80	13.70	13.70
4	4	100	38.00	0.5	710	.700	3.67	87	39.30	34.10	9.37	9.37
4	8	100	38.00	0.5	700	.325	3.95	80	43.40	34.70	12.60	12.60
5	5	100	39.80	0.5	610	.975	3.45	87	42.45	36.80	7.3	7.3
10	10	100	39.80	0.5	610	.625	3.81	87	43.00	34.40	11.0	11.0
5	5	100	39.30	0.5	670	.950	3.66	84	40.30	34.00	13.45	13.45
6	6	100	39.30	0.5	690	.600	3.27	84	43.80	36.40	9.45	9.45
6	12	100	40.20	0.5	800	.650	5.20	83	43.80	36.40	6.76	6.76
7	7	100	40.20	0.5	550	.290	4.09	84	48.25	38.60	14.14	14.14
8	8	100	41.40	0.5	480	.375	2.76	80	45.55	33.80	11.75	11.75
15	15	100	41.40	0.5	550	1.250	3.08	83	48.30	40.10	14.75	14.75
8	8	100	41.40	0.5	510	1.000	5.50	83	48.30	40.10	14.75	14.75
16	16	100	42.00	0.5	550	1.000	5.50	83	48.30	40.10	14.75	14.75
17	17	100	42.00	0.5	550	1.000	5.50	83	48.30	40.10	14.75	14.75
9	9	100	42.00	0.5	530	.325	1.63	89	44.20	38.30	6.42	6.42
18	18	100	42.00	0.5	530	.325	1.63	89	44.20	38.30	6.42	6.42
19	19	100	35.90	0.5	490	.725	3.55	81	40.20	32.60	9.30	9.30
20	20	100	35.90	0.5	780	.350	2.73	79	41.80	33.00	8.97	8.97
10	10	100	35.90	0.5	780	.350	2.73	79	41.80	33.00	8.97	8.97
21	21	100	41.50	0.5	560	.475	2.61	81	48.25	39.00	6.62	6.62
11	11	100	41.50	0.5	570	.300	1.71	82	48.10	39.40	6.66	6.66
22	22	100	40.20	0.5	570	.600	3.42	84	43.00	36.10	1.20	1.20
23	23	100	40.20	0.5	630	.199	3.11	87	41.20	38.50	4.22	4.22
11	11	100	40.20	0.5	630	.199	3.11	87	41.20	38.50	4.22	4.22
24	24	100	40.20	0.5	630	.199	3.11	87	41.20	38.50	4.22	4.22
13	13	100	36.70	0.5	620	.575	3.56	87	41.20	38.50	4.22	4.22
25	25	100	36.70	0.5	620	.575	3.56	87	41.20	38.50	4.22	4.22
13	13	100	36.70	0.5	610	.200	1.22	88	40.50	33.60	3.00	3.00

TABLE 51.—Data on copper extraction in tests of mixed flotation concentrate.

Roast No.	Leach No.	Sulphuric acid content of solution, percent.	Calcine.		Weight, grams.	Tailing.		Percentage of extraction by tailing assay.	
			Weight, grams.	Copper content, percent.		Copper content.			
						Per cent.	Grams.	From calcine.	From original material.
1	1	0.5	100	1.37	88	1.59	1.40	(a)	(a)
1	2	0.5	100	1.37	88	1.59	1.40	(a)	(a)
2	3	0.5	100	1.48	83	1.70	1.41	4.74	4.42
2	4		100	1.48	89	1.69	1.50	(a)	(a)
3	5	0.5	100	1.37	83	1.48	1.21	11.75	10.35
3	6		100	1.37	88	1.48	1.31	4.37	3.86
4	7	0.5	100	1.37	83	1.59	1.32	3.65	3.34
4	8		100	1.37	87	1.48	1.25	9.75	8.00
5	9	0.5	100	1.53	80	1.77	1.42	7.75	7.65
5	10		100	1.53	87	1.58	1.37	10.40	10.35
6	11	0.5	100	1.52	80	1.05	.84	44.60	43.60
6	12		100	1.52	84	1.82	1.53	(a)	(a)
7	13	0.5	100	1.61	83	1.95	1.62	(a)	(a)
7	14		100	1.61	89	1.82	1.62	(a)	(a)
8	15	0.5	100	1.61	80	2.00	1.60	(a)	(a)
8	16		100	1.60	83	1.94	1.61	(a)	(a)
9	17	0.5	100	1.66	80	1.82	1.45	12.65	12.60
9	18		100	1.66	89	1.85	1.65	(a)	(a)
10	19	0.5	100	1.32	81	1.62	1.31	(a)	(a)
10	20		100	1.32	79	1.65	1.30	(a)	(a)
11	21	0.5	100	.95	81	.81	.66	30.60	18.25
11	22		100	.95	82	.82	.67	29.50	17.60
12	23	0.5	100	1.40	84	1.58	1.32	5.72	5.49
12	24		100	1.40	87	1.62	1.41	(a)	(a)
13	25	0.5	100	1.06	85	1.32	1.12	(a)	(a)
13	26		100	1.06	88	1.21	1.07	(a)	(a)

(a) Not analyzed for copper content.

TABLE 52.—Summary of data on extraction of metals in tests of mixed flotation concentrate.

Roast No.	Leach No.	Sulphuric acid content of solution.	Lead extraction.			Zinc extraction.			Copper extraction.		
			Volatilized.	Leached.	Total.	Volatilized.	Leached.	Total.	Volatilized.	Leached.	Total.
			Per ct.	Per ct.	Per ct.	Per ct.	Per ct.	Per ct.	Per ct.	Per ct.	Per ct.
1	1	0.5	36.00	19.65	55.65	5.65	8.90	14.55	15.65		15.65
1	2		36.00	9.50	45.50	5.65	5.20	10.85	15.65		15.65
2	3	0.5	62.50	11.27	73.77	3.58	8.55	11.76	6.95	4.42	11.37
2	4		62.50	6.48	69.98	3.58	2.42	6.00	6.95		6.95
3	5	0.5	21.20	19.00	40.20	4.12	10.26	14.38	11.50	10.35	21.85
3	6		21.20	14.00	34.20	4.12	6.51	10.63	11.50		15.36
4	7	0.5	38.50	28.45	66.95		14.39	14.30	8.30	3.34	11.64
4	8		38.50	25.00	63.50		9.57	9.57	8.30	8.00	16.30
5	9	0.5	51.70	19.30	71.00		13.87	13.87	1.61	7.65	9.26
5	10		51.70	15.02	66.72		8.56	8.56	1.61	10.35	11.96
6	11	0.5	18.90	22.44	41.34	.68	14.25	14.92	2.28	43.60	45.88
6	12		18.90	17.39	36.29	.68	11.23	11.91	2.28		2.28
7	13	0.5	33.70	34.45	68.15	8.85	10.17	19.02	7.45		7.45
7	14		33.70	5.53	39.23	8.85	3.56	12.41	7.45		7.45
8	15	0.5	76.50	8.12	84.62		6.71	6.71	.93		.93
8	16		76.50	2.74	79.24		3.11	3.11	.93		.93
9	17	0.5	23.30	16.85	40.15	.42	13.72	15.34	.26	12.60	12.86
9	18		23.30	11.26	34.56	.42	5.15	5.57	.26		.26
10	19	0.5	34.80	13.17	47.97	3.58	9.22	12.80	9.75		9.75
10	20		34.80	13.55	48.35	3.58	7.56	11.14	9.75		9.75
11	21	0.5	74.50	10.65	85.15		6.16	6.16	40.40	18.25	58.65
11	22		74.50	9.25	85.00		4.59	4.59	40.40	17.60	58.00
12	23	0.5	92.00	9.15	101.15		9.35	9.35	4.24	5.49	9.74
12	24		92.00	8.70	100.70		3.66	3.66	4.25		4.25
13	25	0.5	74.50	9.40	85.47	.017	8.11	8.13	28.60		28.60
13	26		74.50	12.10	81.65	.017	3.16	3.18	28.60		28.60

TABLE 53.—*Data on roasting for tests of electrostatic feed and concentrate.*

[Materials used: No. 1, mixed flotation concentrate; No. 2, feed to electrostatic separator; No. 3, electrostatically treated concentrate.]

Roast No.	Weight of material used.	Weight of calcine used.	NaCl used.	Water used.	Draft, cubic feet water.	Remarks.
	Ounces.	Ounces.	Per ct.	Per ct.	Grains.	
1.....	48	44	6	7	0	No. 2 material used.
2.....	48	43	6	7	1.0	Do.
3.....	48	44	10	6.6	.5	Do.
4.....	48	45	10	6.6	1.0	Do.
5.....	48	45	6	7	.5	39 ounces of No. 2 and 89 ounces of No. 4 material used.
6.....	48	44	6	7	1.0	22 ounces of No. 2 and 14 ounces of No. 4 material used.
7.....	48	45	10	6.6	1.0	32 ounces of No. 2 material and 16 ounces of lead carbonate ore used.
8.....	48	48	10	6.6	.5	Do.
9.....	40	43	10	8	.5	16 ounces of No. 2, 16 ounces of No. 1, and 8 ounces lead carbonate ore used.
10.....	48	41	10		.5	36 ounces of coarsely ground No. 2 material and 12 ounces of No. 2 material (used) ground in ball mill.
11.....	48	46	10	6.6	3.5	Salt and No. 2 material ground in ball mill to pass 100-mesh before roasting.
12.....	48	46	10	6.6	1.5	Do.
13.....	32	31	10	6.6	1.0	Salt and No. 2 material ground in ball mill. Thirty added charge.
14.....	48	46	10	6.6	1.5	Salt and No. 3 material, ground in ball mill.
15.....	32	28	10	6.6	1.0	Do.

TABLE 54.—Volatilization data for tests of electrostatic feed and concentrate.

Roast No.	Lead.			Zinc.			Copper.		
	Content in heading.		Percent- age vola- tilized.	Content in heading.		Percent- age vola- tilized.	Content in heading.		Percent- age vola- tilized.
	Per cent.	Ounces.		Per cent.	Ounces.		Per cent.	Ounces.	
1.....	9.66	4.65	59.70	32.67	15.70		1.58	0.755	1.88
2.....	9.66	4.65	47.40	32.67	15.70		1.58	0.731	1.83
3.....	9.66	4.65	77.50	32.67	15.70		1.58	.642	18.85
4.....	9.66	4.65	75.00	32.67	15.70		1.58	.745	
5.....		4.81	41.00		16.26			.757	
6.....		3.32	2.84		16.81			.770	
7.....		3.32	70.40	32.67	10.50		1.58	.474	5.82
8.....		2.30	74.20		10.50		1.58	.505	30
9.....		3.45	58.40	32.67	11.57		1.58	.491	2.39
10.....		6.32	28.60		10.50			.603	10.75
11.....		3.86	64.50	32.67	15.65			.745	
12.....	9.66	4.65	60.20	32.67	15.65	7.35	1.58	.745	750
13.....	9.66	4.65	68.40	32.67	15.65	6.71	1.58	.745	
14.....	9.66	3.09	49.20	32.67	10.45	3.50	1.58	.505	(a)
15.....	9.18	2.94	82.70	42.60	20.40	2.45	(a)	(a)	(a)
.....	9.18	2.52	76.70	42.60	13.61	4.47	(a)	(a)	(a)
.....				46.60	13.00				

^a Copper not determined.

TABLE 55.—Data on lead extraction in leaching tests of electrostatic feed and concentrate.

Roast No.	Leach No.	Calcine.		Sulphuric acid content, per cent.	Volume, c. c.	Solution.			Tailing.					
		Weight, grams.	Lead content, per cent.			Lead content, grams.	Percentage of extraction—		Weight, grams.	Lead content.		Percentage of extraction—		
							From calcine.	From ore.		Per cent.	Grams.			
													Per cent.	Grams.
1	1	100	4.24	0.5	540	0.250	1.40	33.10	13.30	86	3.94	3.39	26.50	8.25
2	2	100	4.24		490	0.317	1.55	36.60	14.70	84	3.7	3.28	25.80	9.55
3	3	100	5.68	.5	520	.426	2.21	39.00	20.50	83	4.16	3.70	34.80	7.50
4	4	100	5.68		470	.334	1.57	27.61	14.55	87	4.86	4.22	31.20	16.40
5	5	100	2.38	.5	565	1.115	6.65	27.30	6.67	81				
6	6	100	2.38		500	.622	3.46	19.55	4.46	87				
7	7	100	2.58	.5	610	.127	.76	30.00	7.50					
8	8	100	2.58		500	.291	1.45	56.50	11.10					
9	9	100	6.31	.5	550	.542	3.00	37.00	28.10	78	4.15	3.24	18.60	4.50
10	10	100	6.31		490	.426	2.09	33.10	10.55	82	4.15	4.25	32.92	16.00
11	11	100	3.32	.5	530	.415	2.20	66.40	1.75	79	1.97	1.46	56.10	14.05
12	12	100	3.32		510	.391	1.99	60.00	15.75	81	1.58	1.12	55.45	11.25
13	13	100	2.30	.5	630	.201	1.26	55.00	11.20	81	2.67	1.75	56.90	11.65
14	14	100	2.30		470	.163	.48	21.00	5.44	78	2.99	2.32	21.51	8.80
15	15	100	3.45	.5	490	.316	1.53	44.80	18.65	75	2.90	2.38	42.90	13.00
16	16	100	3.45		490	.267	1.01	29.40	12.21	74	4.32	3.22	31.40	13.80
17	17	100	6.32	.5	480	.701	3.37	53.30	37.90	73	4.60	3.54	41.00	13.00
18	18	100	6.32		500	.425	2.12	33.60	23.61	74	2.42	1.79	36.40	13.80
19	19	100	4.65	.5	490	.600	2.69	57.80	25.70	74	3.45	2.62	41.44	14.80
20	20	100	4.65		510	.362	1.79	38.00	11.80	76	3.45	2.62	31.80	13.80
21	21	100	4.02	.5	510	.247	1.26	31.40	12.50	81	3.10	2.32	45.30	11.80
22	22	100	4.02		485	.155	.75	18.65	7.42	74	2.30	1.79	45.30	11.80
23	23	100	3.10	.5	500	.247	1.24	39.40	11.95	75	1.80	1.35	35.30	12.50
24	24	100	3.20		410	.437	1.79	55.70	16.90	79	4.72	3.65	23.40	12.50
25	25	100	5.07	.5	485	.224	1.08	21.00	10.90	81	3.05	2.32	19.40	9.81
26	26	100	5.07		520	.195	.57	29.00	10.15	81	3.05	2.32	19.40	9.81
27	27	100	5.80	.5	430	.086	.37	46.45	6.00	74			16.10	8.00
28	28	100	5.80		300	.161	.66	32.45	6.77	81	1.60	1.20	63.80	15.00
29	29	100	2.32	.5	410	.570	2.34	92.75	12.60					
30	30	100	2.32		370	.402	1.49	50.00	11.60					

a Roasts 14 and 15 were on material No. 3, electrostatic zinc concentrate.

TABLE 56.—Data on zinc extraction in leaching tests of electrostatic feed and concentrate.

Roast No.	Leach No.	Calamine.		Sulphuric acid content, per cent.	Volume, c. c.	Solution.		Tailing.					
		Weight, grams.	Zinc content, per cent.			Zinc content.		Weight, grams.	Zinc content.		Percentage of ex- traction—		
						Per cent.	(grams).		Per cent.	Grams.	From calamine.	From original material.	
1	1	100	37.60	0.5	540	0.595	3.22	8.56	86	40.30	34.80	7.72	7.72
2	2	100	37.60	.5	490	.940	4.61	12.21	84	38.30	32.20	14.35	14.35
3	3	100	37.90		520	.892	4.64	12.23	83	41.00	33.00	12.90	12.90
4	4	100	37.90		470	.595	2.89	7.37	87	39.50	33.40	11.85	11.85
5	5	100	35.50	.5	565	.545	3.08	8.67	81	40.80	33.90	7.05	7.05
6	6	100	35.50		500	.198	.99	3.79	87	42.00	36.60		
7	7	100	36.80	.5	610	.545	3.32	9.05	78	40.80	31.90	13.30	13.30
8	8	100	36.80		500	.745	3.73	10.10	78	40.80	31.40	14.70	14.70
9	9	100	38.20	.5	550	1.190	6.55	17.10	78	39.00	30.41	20.40	20.40
10	10	100	38.20		490	.940	4.61	12.10	82	43.00	35.30	7.07	7.07
11	11	100	40.20	.5	530	1.400	7.40	18.50	74	45.80	33.00	17.90	17.90
12	12	100	40.20		510	1.050	5.30	13.30	79	42.30	33.20	17.40	17.40
13	13	100	24.90	.5	630	.535	3.38	13.50	81	25.00	20.10	19.30	19.30
14	14	100	24.90		470	.248	1.17	4.70	86	26.78	23.00	7.65	7.65
15	15	100	26.40	.5	490	.894	4.37	16.60	78				
16	16	100	26.40		471	.471	2.31	8.74	92				
17	17	100	28.00	.5	480	1.290	6.21	22.10	75	27.21	20.40	28.20	28.20
18	18	100	28.00		500	.595	2.98	10.60	77	27.75	22.90	18.20	18.20
19	19	100	35.50	.5	490	1.140	5.60	15.75	74	38.60	28.60	19.40	19.40
20	20	100	35.50		510	.595	3.03	8.55	77	39.62	30.60	13.50	13.50
21	21	100	31.80	.5	485	.198	.97	3.04	76	37.20	28.20	11.30	10.95
22	22	100	31.80		510	.321	1.60	4.88	81	38.00	30.60	3.78	3.66
23	23	100	32.90	.5	500	.148	.60	1.85	75	42.40	31.40	4.56	4.50
24	24	100	32.90		410	.272	1.32	3.90	74	42.90	32.10	2.43	2.34
25	25	100	33.80	.5	485	.124	.64	1.89	79	41.00	33.20	1.77	1.75
26	26	100	33.80		520	.222	.95	2.20	81	41.00	42.25	2.55	2.15
27	27	100	43.20	.5	430	.222	.95	2.20	74	57.00	42.30	2.03	2.03
28	28	100	43.20		360	.247	.89	2.05	74	57.15	45.50	2.36	2.25
29	29	100	46.60	.5	410	.223	.91	1.99	80	57.10	46.00	1.28	1.28
30	30	100	46.60		370	.124	.46	.99	83	53.50	46.00		.94

TABLE 57. — *Data on copper extraction for leach materials of electrostatic feed and concentrate.*

Roast No.	Leach No.	Calcine.		Sulphuric acid, per cent.	Weight, grams.	Treatment		Percentage of extraction (by tailing assay).	
		Weight, grams.	Copper content, per cent.			Copper content		From calcine.	From original material.
						Per cent.	Grains.		
1.....	1	100	1.70	0.5	83	1.85	1.68	1.17	1.17
1.....	2	100	1.70		84	1.95	1.64	3.34	3.34
2.....	3	100	1.70	0.5	83	1.95	1.61	5.30	5.30
2.....	4	100	1.70		87	1.95	1.70		
3.....	5	100	1.46	0.5	81	1.85	1.50		
3.....	6	100	1.46		87	1.75	1.52		
4.....	7	100	1.66	0.5	78	2.05	1.60	3.52	3.52
4.....	8	100	1.66		78	1.95	1.52	8.60	8.60
5.....	9	100	1.75	0.5	78	2.05	1.60	8.60	8.60
5.....	10	100	1.75		82	1.95	1.60	8.60	8.60
6.....	11	100	1.75	0.5	74	1.95	1.44	17.70	17.70
6.....	12	100	1.75		74	1.95	1.44	12.00	12.00
7.....	13	100	1.05	0.5	81	1.95	1.774	24.20	24.20
7.....	14	100	1.05		86	1.08	1.04	1.32	10.65
8.....	15	100	1.05	0.5	78	1.01	1.79	24.80	24.70
8.....	16	100	1.05		92	1.05	1.00	7.90	7.86
9.....	17	100	1.14	0.5	75	1.53	1.15		
9.....	18	100	1.14		77	1.55	1.20		
10.....	19	100	1.62	0.5	74	1.90	1.41	13.00	13.00
10.....	20	100	1.62		77	1.92	1.48	8.60	7.71
11.....	21	100	1.64	0.5	76	1.83	1.39	15.20	15.20
11.....	22	100	1.64		81	1.83	1.48	7.75	7.75
12.....	23	100	1.63	0.5	74	2.02	1.49	8.60	8.60
12.....	24	100	1.63		75	2.02	1.52	6.75	6.75
13.....	25	100	1.64	0.5	79	2.02	1.59	3.05	3.05
13.....	26	100	1.64		81	2.02	1.63		
14.....	27	100							

TABLE 58. — *Summary of data on extraction of metals in tests of electrostatic feed and concentrate.*

Roast No.	Leach No.	Sulphuric acid content of solution.	Lead extraction.			Zinc extraction.			Copper extraction.		
			Volatilized.	Leached.		Volatilized.	Leached.		Volatilized.	Leached.	
				Per ct.	Per ct.		Per ct.	Per ct.		Per ct.	Per ct.
1.....	1	0.5	59.70	10.77	70.47	8.14	8.14	1.17	1.17	1.17	1.17
1.....	2		59.70	11.63	71.33	13.78	13.78	3.53	3.53	3.53	3.53
2.....	3	0.5	47.40	19.40	66.80	12.56	12.56	1.88	5.20	7.08	7.08
2.....	4		47.40	15.47	62.87	9.61	9.61	1.88		1.88	1.88
3.....	5	0.5	77.50	6.07	83.57	7.86	7.86	18.85		18.85	18.85
3.....	6		77.50	4.36	81.86	3.79	3.79	18.85		18.85	18.85
4.....	7	0.5	75.00	7.50	82.50	11.17	11.17		3.62	3.62	3.62
4.....	8		75.00	14.10	89.10	12.40	12.40	8.40	8.40	8.40	8.40
5.....	9	0.5	41.00	28.40	69.40	18.75	18.75	8.60	8.60	8.60	8.60
5.....	10		41.00	19.43	60.43	9.58	18.20	8.60		8.60	8.60
6.....	11	0.5	70.40	18.12	88.52	15.35	15.35	17.70	17.70	17.70	17.70
6.....	12		70.40	16.37	86.77	16.40	16.40	12.00	12.00	12.00	12.00
7.....	13	0.5	74.20	13.72	87.92	16.40	16.40	30.22	30.22	30.22	30.22
7.....	14		74.20	6.07	80.27	6.17	6.17	10.65	10.65	10.65	10.65
8.....	15	0.5	58.40	16.50	74.90	16.00	16.00	23.70	23.70	23.70	23.70
8.....	16		58.40	10.55	68.95	8.74	8.74	7.86	7.86	7.86	7.86
9.....	17	0.5	29.60	36.00	65.60	26.15	25.15	2.39	2.39	2.39	2.39
9.....	18		29.60	27.30	56.90	14.40	14.40	2.39	2.39	2.39	2.39
10.....	19	0.5	64.50	24.04	88.54	17.57	24.52	10.75	11.60	22.35	22.35
10.....	20		64.50	14.35	78.85	11.02	18.37	10.75	7.70	18.45	18.45
11.....	21	0.5	60.20	13.15	73.35	10.05	16.76	15.20	15.20	15.20	15.20
11.....	22		60.20	7.18	67.38	3.20	10.04	9.75	9.75	9.75	9.75
12.....	23	0.5	69.40	12.82	82.22	6.53	10.03	8.60	8.60	8.60	8.60
12.....	24		69.40	17.05	86.45	3.06	5.56	6.75	6.75	6.75	6.75
13.....	25	0.5	49.20	11.70	60.90	3.96	3.96	3.95	3.95	3.95	3.95
13.....	26		49.20	9.98	59.18	1.81	1.81				
14.....	27	0.5	82.00	5.59	87.59	2.15	4.60				
14.....	28		82.00	6.67	88.67	2.44	4.45				
15.....	29	0.5	76.70	22.60	99.30	2.07	6.54				
15.....	30		76.70	14.80	91.50	.92	5.39				

a Copper not determined.

DISCUSSION OF TESTS WITH FLOTATION CONCENTRATE.

In the tests of flotation concentrate, Table 52 shows that the only satisfactory total extractions of lead were in those roasts that were hot enough to cause considerable volatilization of lead and that rarely was a high extraction of lead obtained by chloridizing roasting followed by leaching with neutral brine or acid brine. Sublimation was the only promising method, as far as the recovery of lead was concerned.

EFFECTS OF TEMPERATURE AND DRAFT.

Tests 1 and 2 (Tables 47 to 52) were designed to be run at a fairly high temperature, hence a rather strong draft suction (2 inch on the water gage) was used. The charge used in test 1 contained 6 per cent of common salt and the one used in test 2 contained 10 per cent salt (see Table 47). Tests 3 and 4 were run to parallel the first two tests, except that the draft was reduced to one-half inch water gage in order that the temperatures might be lower. In each pair of tests the higher percentage of salt gave higher extractions of the lead, and in leaching the calcine a larger proportion of lead was dissolved with acid brine than with neutral brine. The charges used in tests 5 and 6 contained some powdered coal in addition to the natural sulphur content. The higher temperatures resulting did not seem to give any higher extractions of the lead than in the previous tests. Hence test 7 was run with 0.5 inch draft and 6 per cent salt and test 8 with 2 inches draft and 10 per cent salt, to verify the former tests. The results seemed to indicate that with a high draft (high temperature) and a large percentage of salt in the charge more of the lead was volatilized and the total extraction was greater. Therefore, tests 9 and 10 were run with still larger proportions of salt, additional fuel, and high draft in the roaster, with the expectation of obtaining more complete volatilization of the lead. However, the results were not so good as in the former tests.

EFFECTS FROM GRINDING AND MIXING.

In order to determine whether thorough mixing of the salt with the material might improve the reaction, in tests 11, 12, and 13 the material was slightly ground with iron balls in a ball mill with an excess of saturated salt solution, in order to allow intimate mixing of the salt and the mineral particles. The mixture, after being drained dry enough to permit charging into the roaster, was roasted. The excellence of the results shows the importance of thorough mixing. The best way of obtaining a high extraction of lead from material containing mixed sulphides seems to be to grind it fine, mix intimately with about 7.5 per cent salt, and roast in a blast roaster at such a temperature that the lead chloride formed will

be volatilized. A fluffy consistence of the charge in the roaster permits a light draft and good extractions.

DISCUSSION OF TESTS OF ELECTROSTATIC SEPARATOR CONCENTRATES.

The results obtained in the tests of material from the electrostatic concentrator, presented in Tables 53 to 58, which was coarser than the flotation concentrate used in the previous tests, confirm these conclusions. Tests 1 and 2, compared with tests 3 and 4, show that the use of 10 per cent NaCl in the charge gives higher extractions of the lead than 6 per cent. The amount of draft used did not seem to make a great deal of difference in these tests. In tests 5 and 6 some of the finely ground flotation concentrate used in the first series of tests (Tables 47 to 52) was mixed with the coarser concentrate, designated as No. 2 material, Table 53, in order to test the effect of enough slimes being present in the charge to cause balling. Considering that less salt was used than in tests 3 and 4, the high extraction in test 6 shows that a better roast was obtained. In test 5 the draft was too low to give a good roast.

ADDITION OF FINELY GROUND MATERIAL.

In tests 7 and 8 the coarse No. 2 concentrate was made to ball by adding some finely ground lead-carbonate ore. In test 8 the draft was too low, but test 7 showed a good extraction of the lead. In test 9 a mixture of coarse No. 2 concentrate, some flotation concentrate, and some finely ground lead carbonate ore was roasted but the draft used was too low.

FINE GRINDING AND THOROUGH MIXING.

Tests 10 to 15 were made to determine whether fine grinding and intimate mixing would improve the roast. In general the results show that some benefit is derived from this treatment. As much as 90 per cent of the lead can be volatilized and leached from the material. In test 11 the charge settled firmly on the grate, owing to improper charging, and a high draft was necessary in order to get any air through. In this instance the high draft does not mean high temperature.

BALLING.

The finely ground material could be balled with water into a mass that would dry to a fluffy consistence during roasting in the blast roaster, leaving room for an excellent penetration of the gases. It is difficult to so mix coarse concentrates containing no slimes that they will go onto the roaster in such a fluffy mass, which permits roasting under a low draft. A part or the whole of the coarse concentrate can be ground finely to provide this slime. Grinding all

of the charge in saturated brine in a ball mill would insure thorough mixing with the necessary salt, but the subsequent filtering and partial drying of the ground material would be rather expensive, although not excessively so. The better practice would probably be to use some flotation concentrate to ball the coarser particles of sulphide and apply the salt as a saturated brine.

RECOVERY OF ZINC.

In the later tests, in which the salt was mixed with the charge by fine grinding, very little of the zinc in the ore was either volatilized or leached, showing that most of the zinc sulphide was unaffected by the roasting of the lead compounds. The highest percentage of the total zinc content volatilized in any of the tests was 7.35 per cent and the highest percentage leached was 25.15 per cent. The average percentage of total zinc volatilized was less than 5 per cent and the average amount leached was about 8 per cent, hence the average loss of zinc from the concentrate during the removal of the lead was 13 per cent. Of course the greater portion of this zinc would later be recovered from the fume and solutions in the form of zinc oxide, so that it need not be lost. It is a remarkable fact that nearly all the lead could be volatilized and leached from a mixed sulphide ore without markedly affecting the sphalerite in any way.

As regards improvement of grade of the zinc sulphide concentrate by removal of the lead and the partial roasting of the iron sulphide, it can be seen that the average zinc content in the leached residue of the first four tests is 40 per cent, whereas the material before roasting contained 32.67 per cent zinc. It so happens that material containing 32 per cent zinc is difficult to market as zinc concentrate, whereas in many of the contracts for the complex western zinc sulphide concentrates a 40 per cent zinc concentrate is used as the basis on which the buying price is calculated. Often zinc smelters refuse to smelt zinc sulphide ore carrying less than 35 per cent zinc. Therefore, although the resulting zinc sulphide residue is not of a grade to compare with the concentrates from such districts as the Joplin district, because of the large amount of iron pyrite intergrown with the zinc sulphide, this method applied to the ore will effect practically complete separation of the lead and raise the grade of the zinc concentrate from a product of doubtful commercial value to one which could be easily sold and is more desirable to zinc smelters. As the lead in such zinc concentrates is usually not paid for by the zinc smelter, such a saving is of value in conserving resources as well as adding to the possible profits of the shipper of the zinc concentrates. Roasts 14 and 15 were with zinc concentrates from the electrostatic separator in the Mary Murphy mill,

designated as No. 3 material in the tables. Before treatment these concentrates contained 42.6 per cent zinc and 9.18 per cent lead. The treatment removed 90 per cent of the lead and left a residue containing 94 per cent of the zinc as a concentrate containing 57 per cent zinc. The improvement in the grade of this product is even more noticeable.

RECOVERY OF COPPER AND SILVER.

The extraction of the copper and the silver from such zinc concentrates is of interest, as they are lost along with the lead in ordinary methods of zinc smelting, unless the zinc smelter residue is sent to the lead blast furnaces. An inspection of the tables will show that in most of the tests the extractions of copper are practically negligible. As much as 60 per cent of the silver could be dissolved out by quick leaching, but on standing in contact with the zinc sulphide in the calcine from the volatilizing operation to recover the lead, the brine would lose its dissolved silver. Some tests with the above-mentioned calcines on brines containing dissolved copper and silver chlorides showed that the calcine precipitated all of the silver and copper in solution in 16 hours, and about 50 per cent of the silver and copper in 20 minutes. If the brine was strongly acid, precipitation was not so rapid nor nearly so complete. Roasting evidently changed the properties of the material, as far as precipitation of silver and copper from brine solutions are concerned. The raw material did not act like the calcine in this respect, as the precipitation of silver and of copper was very small. Nevertheless, the roasted material seemed to contain zinc sulphide and very little zinc oxide. It was susceptible of flotation, yielding a dark colored blende concentrate, and acted in every way like zinc sulphide.

Also, the calcine tended to reprecipitate lead from the brine. However, most of the lead could be recovered without much trouble, whereas the silver and copper extractions were always low. The action of zinc oxide on the solutions was practically identical with that of the roasted calcines, which were supposedly zinc sulphide. Hence it does not seem possible to make commercial extractions of the silver and copper in zinc-sulphide concentrates, even when the silver does not accompany the zinc sulphide in intimate crystallization.

LIMITATIONS OF THE METHOD.

This latter fact shows that the process is hardly all that could be desired. Its use insures recovery of the lead from such materials, with a considerable increase of the grade of the zinc concentrate, but yields a low recovery of the silver and copper that may be

present. Copper is not an uncommon constituent of such concentrates, although it is not a usual constituent in the bulk of the present tonnage of leady zinc concentrates being sent to the zinc smelters from the intermountain regions. However, silver is usually present in such materials in proportions varying from small amounts to as much as 20 ounces per ton. Where the usual extraction of the silver may be as much as 25 to 33 per cent, it can be seen that the process is not desirable for the treatment of high silver ores. For the treatment of low silver ores and for ores that do not contain silver, this method has much to recommend it. For instance, the great bulk of zinc concentrates shipped from the western mountain States, excluding those from the Butte mines, contain not over 3 ounces per ton of silver. Usually the lead content of such concentrates falls below 5 per cent, in the improved ore-dressing practice of the present time, although considerable quantities contain 10 per cent or more of lead, which is a loss to the companies shipping the zinc concentrate. For such conditions the process is certainly a step in the right direction.

CONCLUSIONS AS TO EXPERIMENTS WITH LEADY ZINC-SULPHIDE CONCENTRATES.

The treatment of leady zinc-sulphide concentrates by chloridizing roasting, followed by brine leaching, gives the following results:

1. It is possible to recover 90 per cent or more of the lead in such concentrates by volatilizing most of the lead in a fairly hot roast on a down-draft shallow-bed roaster, such as the Dwight-Lloyd or the Knight-Christensen roaster, followed by a quick leach with saturated brine acidified with sulphuric acid.

2. For treating coarse concentrates it is advisable to add some finely divided flotation concentrate to the roaster charge in order that the mass may be made to ball with a small amount of water and leave the charge porous during roasting.

3. Six to ten per cent of sodium chloride, based on the weight of concentrate, is necessary for good chloridizing with the average concentrate.

4. The draft necessary to roast a charge bedded 1.5 inches in less than 15 minutes is about 1 inch water gage.

5. The zinc sulphide in the charge is practically unaffected by the combustion of the galena and the pyrite and remains in the residue from leaching. About 90 to 95 per cent of the zinc is recovered in this semisintered residue, which contains a much higher percentage of zinc than the original concentrate treated. Such material should be more acceptable to the zinc smelter because of the absence of lead and its higher grade, assuming that the chloridizing treatment has not injured its roasting properties. In roasting flotation concentrates

the semisintering action binds the small particles into larger aggregates, which should not form so much flue dust on roasting in the zinc smelter.

6. If the material contains silver, usually a recovery of about 30 per cent can be expected, depending on the length of time that the leaching solution is in contact with the calcine. The calcine of zinc sulphide tends to reprecipitate the silver from solution, the speed of precipitation being dependent on the acidity of the solution; hence highly acid solutions permit better extraction of the silver than weakly acid solutions.

7. If copper is present, only 10 to 15 per cent of that metal can be recovered. The zinc sulphide in the residue reprecipitates the copper from solution on leaching. Hence this process is not advisable for the treatment of zinc concentrates containing copper minerals.

8. The costs of treating leady zinc-sulphide materials by this method would probably be about the same as those calculated for oxidized ores. The lead content in the average zinc concentrate should be extracted at a complete metallurgical cost of \$2 to \$2.50 per ton of material. With lead at 4 cents, the normal market price, the lower limit of profitable treatment would be material containing about 4 to 5 per cent lead. Unless material containing less than 4 per cent lead is subject to a penalty at the zinc smelter (as it is in the Joplin district), the bonus paid for the higher zinc content of the concentrate would more than balance the loss of weight and of zinc incurred in the process. It is not probable that any lower grade of material could be worked by this process.

LEAD-IRON MIDDLEINGS AND COMPLEX SULPHIDES.^a

As mentioned earlier in this report, some mixtures of galena and pyrite are occasionally regarded as being too complex for separation and not desirable for shipment to the lead smelters. Usually the iron in the concentrates, after roasting, enters the slag in the lead blast furnace by combining with the silica in the charge. Where only high-grade galena or other lead products are desired, as in the smelting of ores for lead only, or where the smelter is receiving a preponderance of base ores over siliceous ores, or where the cost of shipping the lead-iron mixture to the smelter is too high, it would be desirable to remove this lead by some cheap, simple process. Hence the attempt to apply chloridizing roasting and leaching to this type of product.

^a Experimenter: M. J. Udy.

CHARACTER OF MIDDINGS.

In the disseminated lead district of southeastern Missouri about 50 to 100 tons daily of a lead-iron middling is made in a number of mills, which average 10 to 15 tons each of this product. Part of the lead can be recovered by tabling, but much of it is bound up in a true middling, the treatment of which has presented a difficult problem. Occasionally such middling will contain important amounts of zinc and copper, as indicated by the analyses shown in Table 59 of five different samples received from three mills at different times. In one mill, not represented in the table, the middling is receiving a combined magnetic and table treatment in the attempt to make a zinc concentrate and also a lead concentrate containing less iron. However, it has never yielded as good results as might be desired.

TABLE 59.—*Analyses of lead-iron middlings from mills in southeastern Missouri.*

Sample No.	Source of sample.	Insoluble.	Fe.	Zn.	Pb.	Cu.	S.
		<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
1.....	Bonne Terre mill.....	1.89	27.1	4.5	13.13	1.6	
2.....	do.....	1.00	31.0	3.0	9.2	1.43	34.2
3.....	Leadwood mill.....	2.72	7.1	9.61	41.25	None.	15.1
4.....	do.....	2.41	12.0	26.0	9.75	None.	23.5
5.....	St. Francois mill.....	3.60	11.6	15.0	27.75		18.0

Also some ores may be difficult to treat by ordinary ore-dressing methods, owing to partial oxidation of the ore or to intercrystallization of the various sulphides. Analyses of five materials of this type are given in Table 60. The Bingham Mines Co. ore and the material from the dump of North Star mine are examples of partly oxidized materials that do not give good separation in gravity concentration. The sample from the Bullion Coalition mine is representative of a type of ore so high in iron sulphide that it is difficult to obtain clean lead concentrate and still more difficult to obtain clean zinc concentrate. The ore samples from the Constitution and the Douglas mines represent truly complex microcrystalline ores that yield to no existing ore-dressing method, except that the gangue can be partly removed by fine grinding and flotation. The problems presented by the materials whose analyses are contained in this table are of exceptional magnitude. For 60 years advances have been made in the treatment of complex sulphide ores, but a satisfactory method of treatment is yet to be found. The operators of the electrolytic zinc plants that are supposed to treat such types of ores have their troubles, and installation and operating costs are very high.

TABLE 60. — *Analyses of mixed-sulphide materials*

[Analyst, E. P. Barrett.]

Source of material.	In- sol- uble.	CaO	Fe.	Al ₂ O ₃	Zn.	Pb.	Cu.	Ag.	Au.	S.	As.	CO ₂
	<i>Perct.</i>	<i>Perct.</i>	<i>Perct.</i>	<i>Perct.</i>	<i>Perct.</i>	<i>Perct.</i>	<i>Perct.</i>	<i>Perct.</i>	<i>Perct.</i>	<i>Perct.</i>	<i>Perct.</i>	<i>Perct.</i>
Material from Bullion Coal- ition mine (Utah)	18.0	1.1	22.2	14.6	18.1	10.56	9.19	2.65	0.92	30.32	2.42	Perct.
Material from Bingham Mines Co. mine (Utah) . .	56.3	7.3	8.6	6.2	4.5	6.85	.65	8.66	.61	10.8		8.5
Material from North Star mine (Idaho)	35.3	1.2	14.4	10.3	16.6	5.12	None	5.98	.12	18.6		23
Material from Douglas mine (Idaho)	30.4	1.7	9.5	4.9	24.4	9.9	None	5.26	Trace	16.65		
Material from Constitution mine (Idaho)	17.6	5.5	7.4	11.3	32.5	12.5	None	24.82	.66	21.14		

TESTS OF MIDDLEINGS FROM A MISSOURI MILL.

Eight chloridizing roasts of lead-iron middling from the Bonne Terre mill, in Missouri, were made on the thin-bed down-draft roaster of the Knight-Christensen type. The material, being relatively coarse table middling, packed easily. The first three roasts settled onto the grate so tightly during roasting that getting air through the roasting mass was difficult. On that account enough clay (5 per cent) was added to the fourth roast to ball up the charge when moistened, and thus leave it in fluffy condition for roasting. The extraction of lead was 94 per cent, as compared to 75 per cent without clay. In roast 5 a high percentage of salt and a high blast pressure, and no clay, were used with the purpose of volatilizing all of the lead so that leaching would not be necessary. Due to the packing of the charge, the results were no better than on the first three roasts. Roasts 6, 7, and 8 were made with material balled by adding the proper proportion of slime. This slime was from oxidized lead ore from the La Motte mine, in southeastern Missouri. The slime, which contained 2.5 per cent lead in the form of carbonate and was high in iron and alumina, was of claylike consistence. The mixture balled on the roaster in good condition for easy roasting. Extractions of 95 to 100 per cent of the lead were obtained.

RESULTS OF TESTS.

The detailed data on all the tests are not given herein, but a summary of the results is shown in Table 61, following. Each charge was roasted and the percentage of lead, zinc, and copper that volatilized was determined from the weights and analyses of the heading and the calcine. Two samples of the calcine were then leached, one with neutral saturated brine and the other with acidified brine, in order to recover any lead that did not volatilize and to catch any copper that would dissolve. The results show that as much as 80 per cent of the total lead content can be volatilized, hence the quantity of brine necessary for leaching the remaining lead is small. Also, 25

TABLE 61.—Results of tests of middlings from *Bonne Terre mill in southeastern Missouri*.

Roast No.	Leach No.	NaCl added to charge.	Water added to charge.	Air pressure, inches of water.	Sulphuric acid added to leaching solution.	Time of roasting.	Lead.				Zinc.				Copper.		
							Volatilized in roasting, <i>a</i>	Dis-solved in solution, <i>b</i>	Re-maining in tailing, <i>c</i>	Total lead recovered, <i>d</i>	Volatilized in roasting, <i>a</i>	Dis-solved in solution, <i>b</i>	Re-maining in tailing, <i>c</i>	Total zinc recovered, <i>d</i>	Volatilized in roasting, <i>a</i>	Re-covered in leaching, <i>e</i>	Total.
1	1	3	5.5		0.5	16	<i>P. cl.</i> 25.60	<i>P. cl.</i> 31.60	<i>P. cl.</i> 30.10	<i>P. cl.</i> 57.95	<i>P. cl.</i> 17.40	<i>P. cl.</i> 18.40	<i>P. cl.</i> 21.35	<i>P. cl.</i> 37.27	<i>P. cl.</i> 9.50	<i>P. cl.</i> 0.96	<i>P. cl.</i> 10.46
2	2	3	5.5		0	16	25.60	15.70	22.50	44.70	17.10	13.80	12.00	30.00	9.50	1.5	9.95
3	3	6	4.8		.5	16	22.00	57.40	64.70	53.05	9.52	27.10	20.80	33.17	16.90	32.70	19.60
4	4	6	4.8		0	16	22.00	31.80		56.80	9.52	18.20	21.70	28.17	16.90	25.10	12.00
5	5	10	6.3		.5	16	29.00	60.25	57.75	88.00	12.50	26.20	29.70	40.15	12.39	22.80	35.19
6	6	10	6.3		0	16	29.00	40.05	47.75	71.00	12.50		11.00	23.50	12.39	13.00	25.39
7	7	10	6.3		.5	16	61.75	32.60	32.60	94.35	7.92	23.30	24.30	31.72	30.60	20.10	51.00
8	8	10	6.3		0	16	61.75	21.75	25.20	85.20	7.92	23.30	24.30	31.72	30.60	20.10	51.00
9	9	15	7.4		.5	16	32.90	28.00	35.50		5.55	19.10	16.80	23.50	22.10	3.36	25.76
10	10	15	7.4		0	16	32.90			96.50	5.55		6.31	12.26	22.10		
11	11	10	10.0		.5	16	38.70	58.80	56.80			26.90	21.50	24.20		59.10	59.10
12	12	10	10.0		0	16	38.70	45.00	50.30	86.35		21.30	22.00	23.15		40.20	40.20
13	13	6	10.0		.5	16	81.00	16.35	16.35	97.35	7.75	22.50	19.60	28.55		60.50	60.50
14	14	6	10.0		0	16	81.00	16.35	16.35	97.35	7.50	22.50	19.10	28.30		41.00	41.00
15	15	10	6.7	2	.5	16	27.60	66.30	58.80	90.10		22.00	21.30	21.75		59.00	59.00
16	16	10	6.7	2	0	16	27.60	38.00	42.60	67.90		22.00	17.60	19.80		52.10	52.10

^a Determined from weights and analyses of heading and calcine.^c Determined by analysis of tailing.^b Determined by analysis of solution.^d Determined by adding recovery by volatilization and solution.

to 40 per cent of the zinc was extracted. As this zinc is in the solution containing the lead, the problem of proper treatment of the solution is a serious one. Precipitation of lead from such solutions with lime would cause the lead to be accompanied by the zinc. Electrolytic precipitation of the lead from lead-zinc solutions has been tried, as described elsewhere in this report, with the result that the lead was deposited as sponge metal free from zinc. The sponge metal was melted into bars of lead without much difficulty.

As much as 60 per cent of the copper was extracted in test 7, as shown by the analyses of the tailing. The acid leaches always removed more copper than the neutral leaches, in which it was occasionally possible to observe the formation of a white insoluble compound of copper, probably basic cuprous chloride.

No work has been done on the recovery of a zinc product from the middlings tested, although the experience of one company producing them indicates that it can be done. As this company is finding it possible to make a zinc concentrate from similar material, it is not improbable that the roasted, leached tailing could be passed through a magnetic separator, or ground and treated by flotation in order to float the zinc sulphide from the roasted iron mineral.

The excellent results in extracting the lead when the material was balled with a small amount of clay or slimes, and the facts that the process is cheap and yields metallic lead indicate that it should receive further attention by the companies operating in the southeastern Missouri district.

In roasting this material it was noticed that a large amount of elemental sulphur distilled off ahead of the roasting zone and contaminated the lead-chloride fume from the down-draft roaster. Possibly the sulphur could be distilled or otherwise recovered from this fume and sold, thus lessening the cost of treatment by the process. However, no attempt was made to recover the sulphur in the tests of this material, because they were made simultaneously with tests of many other materials. The treatment of a mixed fume of elemental sulphur and lead chloride has not yet received attention at the Salt Lake City station. Hence the methods mentioned in connection with the oxidized ores may require changes.

TESTS OF MATERIAL FROM BULLION COALITION MINE IN UTAH.

As regards mixed-sulphide ores such as are typified by the Bullion Coalition sample, its analysis (Table 60) is much like that of the lead-iron middlings except that the percentage of gangue materials is somewhat higher and the most valuable metal content outside of the lead and zinc was silver. The material being in lumps as mined, could be crushed to any desired mesh. Extensive tests were carried out with this ore, both in the Christensen down-draft roaster and in the Holt-Dern shaft roaster.

Without giving in detail the mass of data collected, the following may be said to be the general results of the tests of this material.

When roasting such pyritiferous material in the Holt-Dern shaft furnace the high sulphur content made it burn too hot when enough air was supplied to sinter it. On that account the column of material, crushed to pass a 12-mesh screen, was roasted with a low blast pressure, nearly eight hours being required for the roasting zone to travel upward through 1 foot of charge. Under these conditions elemental sulphur distilled from the pyrite in the layer above the roasting zone and condensed in the colder layers higher up in the furnace. When the fire came through the top of the charge the large amount of accumulated sulphur distilled out but tended to make the material on top run into a matte. This trouble was less pronounced under low blast pressure with slow roasting than under high blast pressure with fast roasting.

The part of the charge next the walls of the furnace was always loose after roasting, whereas the middle part seemed to be better roasted and stuck together enough to be lifted from the furnace in fairly adherent lumps. In the earlier tests two complete sets of leaching tests were run, one on the cores and the other on the loose outside parts of these roasts. However, the extractions of metals obtained were about the same for the two samples. In the later tests the whole calcine was sampled for leaching.

Five to twenty-five per cent of the lead volatilized during roasting and the rest of the lead was leached. As high as 90 per cent extraction of the lead is possible by "Holt-Dern" roasting and leaching, the highest extraction being obtained by the use of the lowest roasting temperatures. The silver extractions varied from 1.5 per cent with fast roasting to 60 per cent with slow roasting and an acidified brine leach. One serious drawback to using this method is the large amount of zinc that is roasted to soluble form. As much as 35 per cent of the zinc is extracted, largely by the leaching solution, when good conditions for lead extraction are obtained. This would necessitate electrolytic precipitation of the lead from the leaching solutions.

In the Knight-Christensen roaster with shallow bed, more favorable results were obtained, as the troubles from matting of the charge with elemental sulphur were minimized. In most of the roasts in which 10 per cent of sodium chloride was added to the charge, more than 90 per cent of the lead could be recovered and 50 per cent of the silver. The low extraction of silver is one of the greatest disadvantages of the method and makes it applicable only to ores of low silver content. About 25 per cent of the zinc goes into the leaching solution after the material is roasted in this roaster. About one-third of this zinc is in the form of zinc sulphate and zinc chloride; the rest is zinc oxide. As in previous tests, the volatilized lead chloride was

found to be mixed with a considerable amount of elemental sulphur distilled from the pyrite in the charge. In disposing of the sulphur it would probably be just as well to recover it for sale. In a single small crucible test the fume was mixed with lime and fuel. Most of the lead was reduced to metal and calcium chloride slag was formed. The sulphur was distilled and condensed as low-grade elemental sulphur not containing any arsenic or other volatile constituents of the fume. This test was on too small a scale to have any quantitative value.

TEST OF MATERIAL FROM BINGHAM MINE IN UTAH.

The next material tested, that from Bingham, Utah, is partly oxidized and can not be treated successfully by either gravity concentration or by flotation. The zinc content is low so that the ore is being shipped to the smelters under special contract as low-grade material valuable for lead and silver and, to a minor extent, for copper. A metallurgical treatment capable of application at the mine would eliminate freight costs and smelter costs, with a good chance of being a profitable venture.

With the Holt-Dern roaster there was no difficulty in obtaining good results. It was found that the roast could be run as rapidly as desired and with little sintering. The material was ground to pass through a 12-mesh screen. Whenever sintering did take place the sinter was soft and was readily broken and leached. Ninety-five per cent of the lead and 60 per cent of the silver could be dissolved without difficulty, but only 20 per cent of the zinc was extracted; as the material was low in zinc, the proportion of that metal entering the solution was so small that precipitation of the lead with lime was permissible. Only a small proportion of the lead extracted was volatilized; most of it was obtained from the leaching solution. Seemingly, the character of the gangue makes this material easy to treat by this method and permits high extractions. The results are an example of the great difference in physical and chemical characteristics of different ores, as the other materials tested previously had been rather hard to treat successfully. It would also seem, from observation of the roasts, that the content of sulphur in this ore was about the largest amount that can be used to make the ore self-roasting, and yet not cause sintering.

Roasting the ore in the Knight-Christensen down-draft roaster was even more successful as regards the recovery of silver; a number of tests showed an 80 per cent extraction. When roasting was followed by an acid-brine leach, the lead extractions were uniformly higher than 90 per cent. About a third of the zinc was dissolved by the leaching solution. The lead obtained in fume by volatilization never amounted to more than 30 per cent of the total lead content.

From these tests one can conclude that when the ore treated does not contain too much sphalerite and pyrite, the roasting is easy, and high extractions of both the lead and the silver are possible. Hence, it would seemingly be better to remove the lead and the silver from the ore by this method before the zinc sulphide is concentrated rather than to try to remove the lead from a complex of the two sulphides concentrated from the gangue. This type of ore most assuredly deserves further attention on the part of those who are now exploiting such materials.

EXPERIMENTS WITH MATERIAL FROM NORTH STAR DUMP, HAILEY, IDAHO.

Tests were also made of the material from the North Star mine dump, near Hailey, Idaho. Contrary to the experience with the Bingham ore, most of the lead (60 per cent) would volatilize during down-draft roasting no matter what the air pressure or the proportion of salt used in the roast. As good extractions of lead and silver could be obtained by using 3 per cent, based on the weight of the ore used, as with 20 per cent of salt. An extraction of about 85 per cent of the lead—60 per cent by volatilization and 25 per cent by leaching—was uniformly possible when the leaching was with acidified brine. In these tests only about 10 per cent of the zinc usually appeared in the leaching solution; the rest was not converted to soluble form and remained in the leached tailing. The ore sinters easily and could thus be spoiled for leaching by careless roasting. An attempt to float the zinc sulphide in the tailing was successful, although the zinc concentrate contained only 42 per cent zinc and the extraction was only 75 per cent. Because of the complex character of this ore, and the fact that the zinc content exceeds the proportion that the lead smelters accept without penalizing, it would seem that removal of the lead and silver by roasting and leaching, followed by flotation of the zinc to form zinc concentrate which would otherwise be lost, should be well worth considering.

MATERIALS FROM MINES NEAR PINE CREEK, IDAHO.

The samples from the Douglas and the Constitution mines, Pine Creek, Idaho, represent material from practically the same ore bodies but from adjoining claims. The Douglas ore is one of the ores being treated in the electrolytic zinc plant of the Anaconda Copper Co. at Great Falls, Mont. The ore from these two mines is so complex that the unaided eye can distinguish no crystals of usually recognizable minerals. The galena, sphalerite, pyrite, and other constituents are so intimately intergrown that the mass is dull black, like manganese ore. Examination of a polished section of this black

aggregate under a high-power microscope reveals the fact that the minerals are all present in distinct types, and are not combined in a "chemical compound," as some persons claim. Under magnification the sphalerite is seen to be yellow and the pyrite has its proper color. There is a great deal of this complex ore throughout the Couer d'Alene district.

In the down-draft roaster, with 6 to 10 per cent salt, the Douglas ore was self-roasting. It is possible to volatilize more than 90 per cent of the lead and leach about 25 per cent of the silver. About 20 per cent of the zinc volatilizes and 5 to 10 per cent is leached. A draft of only one-half inch water gage is sufficient in roasting this ore.

The material from the Constitution mine, on the other hand, could not be treated with any success. It was difficult to volatilize more than 50 per cent of the lead, and only 10 to 20 per cent more could be leached. The cause of this great difference in two ores supposedly from the same ore zone is not known.

COMPARISON WITH ELECTROLYTIC METHOD.

As regards the comparative merits of the method used in those tests and that of making electrolytic zinc from a solution of zinc sulphate leached from "dead-roasted" ore, it is known that the electrolytic treatment does not extract much more than 70 per cent of the zinc, because of the difficulty of converting zinc sulphide to sulphate by roasting. Zinc sulphide is the hardest of the sulphides to roast on a large scale. Consequently in treating such material only enough zinc is removed to permit the residue being smelted in a lead smelter for the silver, gold, and lead. In the chloridizing and leaching method only the lead, which is easily converted to soluble form, and about one-third of the silver is recovered. The zinc sulphide which remains in the residue can be recovered by flotation, in some ores by shipment of the zinc to the zinc smelter. As regards costs of installation and operation, the chloridizing method of removing lead should have the advantage over the electrolytic zinc process, as long as silver results are disregarded. In treating material with a high-silver content the electrolytic zinc process will have to be used, but where the silver content is low or negligible the chloridizing method is to be preferred on account of its simplicity and cheapness.

In general, it may be said that chloridizing volatilization or chloridizing roasting and leaching does not solve the general problem of treating complex sulphides, but only certain special phases of it.

GENERAL SUMMARY.

1. The application of new hydrometallurgical and other methods has been made to the following ore types:

- (a) Oxidized lead ores.
- (b) Oxidized lead ores containing precious metals.
- (c) Oxidized lead-zinc ores.
- (d) Simple sulphide ores of lead.
- (e) Lead-zinc concentrates.
- (f) Lead-iron sulphide middlings.
- (g) Complex sulphide ores of lead, zinc, iron, and copper, with or without precious metals.

2. It has been found that a saturated solution of sodium chloride, common salt, is a good solvent for lead chloride and lead sulphate and in all of the leaching rests reported herein brine leaches, with or without the addition of sulphuric acid, were used.

3. The lead may be precipitated with lime from solutions that are not contaminated with other elements, and by electrolysis when the solutions are contaminated.

4. Three alternative methods have been tested in the treatment of materials containing oxidized lead, as follows: Sulphidizing and flotation, acid-brine leaching of the raw material, and volatilization of the lead with the recovery of lead chloride fume. All ores do not yield to flotation, and only those that do not contain excessive proportions of acid-consuming constituents yield to acid-brine leaching, whereas nearly all the materials tested have yielded to the volatilization method, irrespective of the kind of gangue.

5. The oxidized ores of lead are usually argentiferous, but occasionally some gold is also present. Silver is often present in considerable amounts as the chloride, which is soluble in acid brine. The sulphide of silver is only slightly soluble in acid brine, so that the acid-brine method of treating oxidized lead ores usually fails to give good extractions of the silver. Sulphidizing and flotation, where applicable, usually extracts the precious metals together with the oxidized lead minerals. However, the experiments described herein have shown that the precious-metal minerals are not necessarily intercrystallized with the lead minerals and usually will be extracted in smaller proportions than is the lead. Chloridizing roasting and leaching give better results, although the best conditions seem to be those where only part of the lead chloride is volatilized, the rest of the lead being leached with the silver and the gold. By raising the temperature of the roast to 900° C. or more, the silver and the gold will be volatilized with the lead, making leaching unnecessary.

6. Treatment of the oxidized zinc-lead ores is more difficult. Sulphidizing and flotation will often remove the lead minerals, together with any silver that might be present, but much of this ore contains

sulphur-consuming constituents that make sulphidizing difficult. The lead can be removed by the chloridizing and volatilization method, although some of the zinc tends to accompany it, using up salt and lowering the extraction of lead.

7. The simple sulphide ores of lead may be treated by the volatilization method, or by chloridizing or sulphating roasting followed by brine leaching. Where silver is present chloridizing roasting and leaching seems to be the most successful method and promises to compete with gravity concentration and flotation, followed by smelting.

8. Lead can be removed from leady zinc sulphide concentrates without affecting the zinc sulphide, by a quick chloridizing and volatilization. The lead chloride fumes can be caught in an electrical precipitator, and converted into metallic lead by fusion with lime and carbon, yielding a calcium chloride slag which is a desirable substitute for salt in chloridizing and volatilization.

9. Lead may be extracted almost completely from lead-iron sulphide middlings by the volatilization method mentioned. Silver, if present, usually does not chloridize well and does not volatilize; also brine leaching gives rather low extractions of silver.

10. Extraction of the lead from the complex mixed sulphides of lead, zinc, copper, iron, and the precious metals by the volatilization method is likewise possible. Very little of the copper, silver, or gold is chloridized in this method and hence, even after chloridizing leaching, these metals remain with the zinc sulphide. The iron sulphide is generally converted to ferric oxide in roasting, giving the material the appearance of having been thoroughly roasted. However, the unaltered sulphides of zinc and copper, and any residual lead sulphide, can usually be removed from the roasted material by fine grinding and flotation, the iron being left in the residue.

11. The methods mentioned cover most of the important places in which lead losses occur. Possibly certain flue dusts from lead smelters could be advantageously treated by brine leaching, as the lead in the flue dust usually consists of basic lead sulphate and lead oxide which are soluble in such a solution. However, no experiments were made with such dusts.

12. Some of the proposed processes, from a study of their probable costs, deserve serious consideration, as they promise to be cheap and usually give higher extractions of metal than are possible by the usual ore-dressing methods. The fact that most of them produce metal, not concentrate, will particularly recommend them to localities distant from smelting or transportation facilities. Also, methods adapted to arid conditions in arid regions are presented that should be of importance in the exploitation of the low-grade lead ores in some western districts.

PUBLICATIONS ON MINERAL TECHNOLOGY.

A limited supply of the following publications of the Bureau of Mines has been printed and is available for free distribution until the edition is exhausted. Requests for all publications can not be granted, and to insure equitable distribution applicants are requested to limit their selection to publications that may be of especial interest to them. Requests for publications should be addressed to the Director, Bureau of Mines.

The Bureau of Mines issues a list showing all its publications available for free distribution as well as those obtainable only from the Superintendent of Documents, Government Printing Office, on payment of the price of printing. Interested persons should apply to the Director, Bureau of Mines, for a copy of the latest list.

PUBLICATIONS AVAILABLE FOR FREE DISTRIBUTION.

BULLETIN 64. The titaniferous iron ores in the United States; their composition and economic value, by J. T. Singewald, jr. 1913. 145 pp., 16 pls., 3 figs.

BULLETIN 70. A preliminary report on uranium, radium, and vanadium, by R. B. Moore and K. L. Kithil. 1913. 100 pp., 2 pls., 2 figs.

BULLETIN 73. Brass furnace practice in the United States, by H. W. Gillett. 1914. 298 pp., 2 pls., 23 figs.

BULLETIN 84. Metallurgical smoke, by C. H. Fulton. 1915. 94 pp., 6 pls., 15 figs.

BULLETIN 85. Analyses of mine and car samples of coal collected in the fiscal years 1911 to 1913, by A. C. Fieldner, H. I. Smith, A. H. Fay, and Samuel Sanford. 1914. 444 pp., 2 figs.

BULLETIN 92. Feldspars of the New England and Northern Appalachian States, by A. S. Watts. 1916. 180 pp., 3 pls., 22 figs.

BULLETIN 103. Mining and concentration of carnotite ores, by Karl L. Kithil and John A. Davis. 1917. 89 pp., 14 pls., 5 figs.

BULLETIN 104. Extraction and recovery of radium, uranium, and vanadium from carnotite, by C. L. Parsons, R. B. Moore, S. C. Lind, and O. C. Schaefer. 1915. 124 pp., 14 pls., 9 figs.

BULLETIN 106. The technology of marble quarrying, by Oliver Bowles. 1916. 174 pp., 12 pls., 33 figs.

BULLETIN 110. Concentration experiments with the siliceous red hematite of the Birmingham District, Alabama, by Joseph T. Singewald, jr. 1917. 91 pp., 1 pl., 47 figs.

BULLETIN 123. Analyses of mine and car samples of coal collected in the fiscal years 1913 to 1916, by A. C. Fieldner, Howard I. Smith, J. W. Paul, and Samuel Sanford. 1918. 456 pp.

BULLETIN 124. Sandstone quarrying in the United States, by Oliver Bowles. 1917. 143 pp., 6 pls., 19 figs.

- BULLETIN 128. Refining and utilization of Georgia kaolins. by I. E. Sproat. 1916. 55 pp., 5 pls., 11 figs.
- BULLETIN 146. Technology of salt making in the United States. by W. C. Phalen. 1917. 149 pp., 24 pls., 10 figs.
- TECHNICAL PAPER 8. Methods of analyzing coal and coke. by F. M. Stanton and A. C. Fieldner. 1913. 42 pp., 12 figs.
- TECHNICAL PAPER 43. The effect of inert gases on inflammable gaseous mixtures. by J. K. Clement. 1913. 24 pp., 1 pl., 8 figs.
- TECHNICAL PAPER 50. Metallurgical coke. by A. W. Belden. 1913. 48 pp., 1 pl., 23 figs.
- TECHNICAL PAPER 76. Notes on the sampling and analysis of coal, by A. C. Fieldner. 1914. 59 pp., 6 figs.
- TECHNICAL PAPER 88. The radium-uranium ratio in carnotites. by S. C. Lind and C. G. Whittemore. 1915. 29 pp., 1 pl., 4 figs.
- TECHNICAL PAPER 95. Mining and milling of lead and zinc ores in the Wisconsin district, Wisconsin. by C. A. Wright. 1915. 39 pp., 2 pls., 5 figs.
- TECHNICAL PAPER 102. Health conservation at steel mills. by J. A. Watkins. 1916. 36 pp.
- TECHNICAL PAPER 110. Monazite, thorium, and mesothorium. by K. L. Kithil. 1915. 32 pp., 1 fig.
- TECHNICAL PAPER 111. Safety in stone quarrying. by Oliver Bowles. 1915. 48 pp., 5 pls., 4 figs.
- TECHNICAL PAPER 126. The casting of clay wares. by T. G. McDougal. 1916. 26 pp., 6 figs.
- TECHNICAL PAPER 136. Safe practice at blast furnaces. by F. H. Willcox. 1916. 73 pp., 1 pl., 43 figs.
- TECHNICAL PAPER 143. Ores of copper, lead, gold, and silver. by C. H. Fulton. 1916. 41 pp.
- TECHNICAL PAPER 155. Gypsum products, their manufacture and uses. by R. W. Stone. 1917. 67 pp.
- TECHNICAL PAPER 177. Preparation of ferrouanium. by H. W. Gillett and E. L. Mack. 1917. 46 pp., 2 figs.
- TECHNICAL PAPER 182. Flotation of chalcopryrite in chalcopryrite-pyrrhotite ores of Southern Oregon. by Will H. Coghill. 1918. 13 pp., 1 fig.
- TECHNICAL PAPER 187. Slag viscosity tables for blast-furnace work. by A. L. Field and P. H. Royster. 1918. 38 pp., 1 fig.
- TECHNICAL PAPER 198. Sulphur dioxide method for determining copper minerals in partly oxidized ores. by Chas. E. van Varneveld and Edmund S. Leaver. 1918. 12 pp.

PUBLICATIONS THAT MAY BE OBTAINED ONLY THROUGH THE SUPER-INTENDENT OF DOCUMENTS.

- BULLETIN 11. The purchase of coal by the Government under specifications, with analyses of coal delivered for the fiscal year 1908-9. by G. S. Pope. 80 pp. 10 cents.
- BULLETIN 12. Apparatus and methods for the sampling and analysis of furnace gases. by J. C. W. Frazer and E. J. Hoffman. 1911. 22 pp., 6 figs. 5 cents.
- BULLETIN 22. Analyses of coals in the United States, with descriptions of mine and field samples collected between July 1, 1904, and June 30, 1910, by N. W. Lord, with chapters by J. A. Holmes, F. M. Stanton, A. C. Fieldner, and Samuel Sanford. 1912. Part I, Analyses, pp. 1-321; Part II, descriptions of samples, pp. 321-4, 129. 85 cents.
- BULLETIN 29. The effect of oxygen in coal, by David White. 80 pp., 3 pls. 20 cents.

BULLETIN 38. The origin of coal, by David White and Reinhardt Thiessen, with a chapter on the formation of peat, by C. A. Davis. 1913. 390 pp., 54 pls. 80 cents.

BULLETIN 41. Government coal purchases under specifications, with analyses for the fiscal year 1909-10, by G. S. Pope, with a chapter on the fuel-inspection laboratory of the Bureau of Mines, by J. D. Davis. 1912. 97 pp., 3 pls., 9 figs. 15 cents.

BULLETIN 42. The sampling and examination of mine gases and natural gas, by G. A. Burrell and F. M. Seibert. 1913. 116 pp., 2 pls., 23 figs. 20 cents.

BULLETIN 47. Notes on mineral wastes, by C. L. Parsons. 1912. 44 pp. 5 cents.

BULLETIN 53. Mining and treatment of feldspar and kaolin in the southern Appalachian region, by A. S. Watts. 1913. 170 pp., 16 pls., 12 figs. 35 cents.

BULLETIN 71. Fullers' earth, by C. L. Parsons. 1913. 38 pp. 5 cents.

BULLETIN 81. The smelting of copper ores in the electric furnace, by D. A. Lyon and R. M. Keeney. 1915. 80 pp., 6 figs. 10 cents.

BULLETIN 107. Prospecting and mining of copper ore at Santa Rita, N. Mex., by D. F. MacDonald and Charles Enzian. 1916. 122 pp., 10 pls., 20 figs. 25 cents.

BULLETIN 108. Melting aluminum chips, by H. W. Gillett and G. M. James. 1916. 88 pp. 10 cents.

BULLETIN 111. Molybdenum; its ores and their concentration, with a discussion of market, prices, and uses, by F. W. Horton. 1916. 132 pp., 18 pls., 2 figs. 30 cents.

BULLETIN 116. Methods of sampling delivered coal, by G. S. Pope. 1916. 64 pp., 5 pls., 2 figs. 15 cents.

BULLETIN 121. The history and development of gold dredging in Montana, by Hennen Jennings, with a chapter on placer-mining methods and operating costs, by Charles Janin. 1916. 64 pp., 29 pls., 1 fig. 30 cents.

BULLETIN 122. The principles and practice of sampling metallic metallurgical materials, with special reference to the sampling of copper bullion, by Edward Keller. 1916. 101 pp., 13 pls., 31 figs.

TECHNICAL PAPER 3. Specifications for the purchase of fuel oil for the Government, with directions for sampling oil and natural gas, by I. C. Allen. 1911. 13 pp. 5 cents.

TECHNICAL PAPER 26. Methods for the determination of the sulphur content of fuels, especially petroleum products, by I. C. Allen and I. W. Robertson. 1912. 13 pp., 1 fig. 5 cents.

TECHNICAL PAPER 41. The mining and treatment of lead and zinc ores in the Joplin district, Mo., a preliminary report, by C. A. Wright. 1913. 43 pp., 5 figs. 5 cents.

TECHNICAL PAPER 60. The approximate melting points of some commercial copper alloys, by H. W. Gillett and A. B. Norton. 1913. 10 pp., 1 fig. 5 cents.

TECHNICAL PAPER 90. Metallurgical treatment of the low-grade and complex ores of Utah, a preliminary report, by D. A. Lyon, R. H. Bradford, S. S. Arentz, O. C. Ralston, and C. L. Larson. 1915. 40 pp. 5 cents.

TECHNICAL PAPER 93. Graphic studies of ultimate analyses of coals, by O. C. Ralston, with a preface by H. C. Porter. 1915. 41 pp., 3 pls., 6 figs. 10 cents.

TECHNICAL PAPER 109. Composition of the natural gas used in 25 cities, with a discussion of the properties of natural gas, by G. A. Burrell and G. G. Oberfell. 1915. 22 pp. 5 cents.

INDEX.

A.		Page	C.		Page
Acid, consumption of, in leaching tests	25, 28, 38		Chlorine, brine leaching of, results of	131, 134	
efficiency of, in leaching tests	24, 25, 29, 31		after in, extraction of	134	
Acid-proof construction of leaching plants	48		Chlorine chloride, leaching point of	50	
Allen, G. L., work of	96		use of in brine leaching tests	33, 127	
American Flag mine (Utah), oxidized ore			Chlorine compound for sulphidizing	103	
from, analysis of	101		Chlorine polysulphide, as sulphidizing agent		
brine leaching test of	24, 25		test of	103	
cyclic leaches of	53		Chloro-Mineral Co., cooperation of	30	
roasting and leaching tests of	63		Chance-Delmonte mine (Utah), oxidized ore		
Augustine process, test of	58, 60		from, analysis of	13	
B.			Chemical reactions in brine leaching	58	
Bacon, R. F., sulphidizing process of	85		Chief Consolidated mine (Utah), oxidized ore		
Bequerel, M. M., roasting and leaching process of	123		from, analysis of	13	
Bingham Mines Co., mine (Utah), oxidized ore from, chloride volatilization of, results of	77		brine leaching tests of	24, 25	
sulphide ore from, analysis of	161		Chief Consolidated mine (Utah), oxidized ore		
roasting tests of	165		from, chloride volatilization of	84	
Bonne Terre mill (Mo.), lead-iron sulphide middlings from, analyses of	160		cyclic leaches of, results of	55	
roasting tests of	161		lead in, association of	15	
Bradford, R. H., work of	9		section analysis of	18	
Brine, acidified, solubility of lead chloride in tests of	23-31		sulphidizing and flotation test of, results of	90	
use of, in tests	14		Chloride of lime, as solvent, in leaching tests	47	
Brine leaching of oxidized ores, chemical reactions in	58		Chloride volatilization, necessary temperatures in	80	
consumption of acid in	26, 41, 42		Chloridizing-leaching process, description of	118	
figure showing	26, 27		Chloridizing roasting, application of	75, 168	
cyclic, description of	34-38		losses in	58	
large scale, description of	38-45		See also Roasting and volatilization.		
results of	49		Christensen, N. C., cooperative work of	67	
time required in	49		Christensen & Holt, chloridizing and leaching process, description of	118	
efficiency of, figure showing	26, 27, 28		Christensen roaster, laboratory size, figure showing	68	
flow sheet of, figure showing	39		tests with	68-72	
methods in	23, 25, 27, 30		Colorado mine (Utah) oxidized ore from, analysis of	13	
results of	31		lead in, association of	15	
use of lime as precipitant in	35, 43		roasting and leaching test of	62	
of calcines, results of	129, 131		Comet mine (Mont.), zinc sulphide concentrate from, analysis of	141	
of sulphide ores, applicability of	169		Concrete tanks, possible use of	49	
See also Leaching.			Constitution mine (Idaho), sulphide ore from, analysis of	161	
Bullion Beck mine (Utah), oxidized slimes from, analysis of	13		treatment of	166	
cyclic leaches of	53		Copper, in lead-iron middlings, extraction of	163	
oxidized tailing from, cyclic leaches of, results of	56		in low-grade ores of, investigations of	9	
sulphidizing test of	99		in oxidized ores, extraction of by chloride volatilization	77, 78, 79	
Bullion Coalition mine (Utah), sulphide ore from, analysis of	161		in oxidized ores, extraction of by sulphidizing and flotation	91	
Bullionville mine (Nev.), oxidized tailing from, analysis of	13		in sulphide concentrate, extraction of by roasting and leaching	148, 153, 157	
cyclic leaches of	53		in zinc sulphide concentrates, volatilization test of, results of	150	
results of	55		Copper chloride, volatilization temperature of	80	
volatilization of	69, 70		Copper Queen Mining Co. mine (Ariz.), oxidized tailings from, analysis of	13	
volatilization and leaching of, results of	73		cyclic leaches of, results of	55	
Bunker Hill & Sullivan Mining & Concentrating Co., brine leaching tests by	31		Cottrell, F. G., acknowledgment to	33	
experimental plant of	123		Crowder, ———, work of	81, 82	
tests at	133		Current density in electrolytic precipitation	51	
Bureau of Mines, cooperative work of	9, 11				

	D.	Page.		Page.
Daily Judge mine (Utah), oxidized ores from,			Gold, volatilization of, reactions in	80
analysis of	13		Great Salt Lake, sodium sulphate in	33
brine leaching test of	25		Grinding, fine, for sulphidizing and flotation,	
lead in, association of	15		effects of	89-92
sulphidizing and flotation tests of, re-				
sults of	90		H.	
zinc sulphide concentrates from, analyses			Handy, R. S., cited	123
of	141		Hayden Development Co. mine (Ariz.),	
Daily West mine (Utah), oxidized tailing			oxidized ore from, analysis of	13
from, analysis of	13		chloride volatilization of, results of ..	78
cyclic leaches of, results of	56		Heating of brine for leaching, effect of	46
Demassieux, N., cited	22		Hercules mill (Idaho), zinc sulphide concen-	
Dorr classifier, use of in brine leaching	108		trate from, analysis of	141
Dorr thickener, use of in brine leaching	109		Highland Surprise mill, zinc sulphide concen-	
Douglas mine (Idaho), sulphide ore from,			trate from, analysis of	141
analysis of	161		Hofman, Ottokar, cited	94
roasting and leaching tests of	166		Holt-Dern roaster, description of	58, 59
Dry Valley mill (Nev.), oxidized tailing from,			figure showing	59
chloride volatilization of, results of ..	77		laboratory size, description of	60
Dry Valley mine (Nev.), oxidized tailing from,			figure showing	61
analysis of	13		mixing of charge for, precautions in ..	61
cyclic leaches of, results of	56		use of in tests	164, 165
volatilization tests of	70, 71		Horn Silver mine (Utah), oxidized tailing	
volatilization and leaching of, results			from, analysis of	13
of	73		brine leaching test of	25
Dwight-Lloyd sintering machine, possible			chloride volatilization of, results of ..	77
use of	115		cyclic leaching tests of	51
			leaching tests of	34-38, 53-56
E.			lead in, association of	15
Electrolytic precipitation in brine leaching,			condition of	36
advantages of	113		screen analysis of	19
cost of	113		Hovland, H. B., sulphidizing process of	86
use of	50-57		Hovland, H. B., and Frankforter, C. B.,	
Eureka Hill mine (Utah), oxidized slime			sulphidizing, process of	85
from, analysis of	13		Hydrochloric acid solution as leach, test of ..	21
cyclic leaches of, results of	55		Hydrogen sulphide, as precipitant in cyclic	
Extraction, maximum, in brine leaching, con-			leaches, use of	44
ditions for	45, 50		cost of	88
<i>See also</i> Copper, gold, lead, silver, zinc.			danger in use of	88
			methods of making	88
F.			sulphidizing tests with	87-93
Filter, laboratory, figure showing	40		Hydrometallurgy of low-grade lead ores,	
Flotation, investigations of	12		methods in	16
of lead carbonate ores	16, 84-106			
conclusions on	122		I.	
concentrate, roasted, leaching tests of,			Iron, corrosion of, in leaching plant	48
results of	146		scrap, as precipitant in cyclic leaches,	
roasting and leaching tests of	144		attempted use of	44
volatilization test of, results of	145		Iron anode, use of in cyclic leaches	51
<i>See also</i> Sulphidizing.			Iron Blossom mine (Utah), oxidized ore from,	
Flotation oils, results of tests with	97-99		analysis of	13
Flow-sheet of brine-leaching plant, descrip-			brine leaching test of	25
tion of	39		lead in, association of	15
figure showing	48, 107		J.	
Flow-sheet of volatilization mill, figure show-			Joplin district (Mo.), zinc blende concentrate	
ing	116		from, analysis of	141
Frisco mill (Idaho), zinc sulphide concentrate				
from, analysis of	141		K.	
Fume from volatilization, treatment of	83		Knight-Christensen mill, brine-leaching test	
			at	60
G.			Knight-Christensen roaster, use of in tests ..	164, 165
Galena, recovery of, from slimes	123			
roasting and leaching of	125		L.	
unroasted, leaching of, tests of	137		La Motte mine (Mo.), oxidized ores from,	
Godiva mine (Utah), oxidized ore from,			analysis of	13
analysis of	13		Laney, F. B., work of	11
Gold, extraction of, by chloride volatilization,	77-79		Larson, C. L., cited	136
by sulphidizing and flotation	97, 99, 100		work of	32, 33, 134
by volatilization and leaching	73		Leaching of unroasted galena, tests of	137

	Page.		Page.
Leaching plant, costs in	110	Metallurgy of ores, problems in	10, 12
cost of	112	Michigan-Utah mine (Utah), oxidized ore	
flow-sheet of, figure showing	48	from, analysis of	13
for oxidized ores, description of	107-110	cyclic leaches of, results of	55, 56
materials for construction of	48	difficulty in sulphidizing and flota-	
probable life of	111	tion of	102
Leaching process, essentials for	112	Mining cost for carbonate ores	111, 121
<i>See also</i> , Brine leaching.			
Lead, carbonate ores of, analyses of	13	N.	
proposed leaching of	107, 112	Nevada United mine (Nev.), oxidized ores	
in lead-iron sulphide middlings, extrac-		from, analysis of	13
tion of	163	chloride volatilization of, results of ..	76
in oxidized ores, extraction of by brine		North Star mine (Idaho), sulphide ore from,	
leaching	24-29, 35, 38, 46, 47	analysis of	161
by chloride volatilization	76-79	roasting and leaching, tests of	166
by chloridizing roast	66		
by cyclic leaches	49, 51, 53, 54, 55, 56	O.	
by roasting and leaching	63	Oils, flotation, tests with	89-91, 97-99
by sulphidizing and flotation	89,	Ontario mill (Utah), test of Augustine pro-	
90, 91, 97, 100, 104		cess at	60
by volatilization	72	Ontario mine (Utah), oxidized tailing from,	
by volatilization and leaching	73	analysis of	13
in sulphide concentrates, extraction of by		cyclic leaches of, results of	55
roasting and leaching	146, 152	Ores, low-grade, investigations of	9
volatilization test of, results of	150	Ores, oxidized, testing of	14, 15
in sulphide ores, extraction of by brine		<i>See also</i> Brine leaching, chloridizing,	
leaching	129, 131	roasting, volatilization, and mines	
by roasting and leaching	164, 166	and mills named.	
smelting of	139	Oxidizing agents, possible use of in volatila-	
metallizing of for flotation	105	tion	81
sulphide ores of, investigations of	123-169		
Lead carbonate, losses of in milling	84	P.	
ores, investigations of	12-122	Pohle-Croasdale process, use of	75
Lead chloride, boiling point of	80	Precipitate, from brine leaching plant, value	
solubility of	20	of	110, 111
figure showing	20	reduction of to metallic lead, advantage	
in brine	22	in	112
thermal decomposition of	66	from cyclic leaches, assays of	42
Lead ores, low-grade, treatment of	16	Prince Consolidated mine (Nev.), oxidized	
<i>See also</i> Brine leaching, chloridizing		ore from, analysis of	13
roasting, roasting and leaching,		volatilization and leaching of, results	
volatilization.		of	73
Lead-iron sulphides, separation of lead in...	160	Pulp, heating of, in leaching tests, effect of...	46
Leadwood mill (Mo.), lead-iron sulphide			
middlings from, analyses of	160	R.	
Lime, as precipitant in brine leaching	114	Roasting, chloridizing, losses in	58
in cyclic leaches	43	temperature conditions in	82
cost of	110	<i>See also</i> Chloridizing roasting, volatila-	
precipitation with, results of	35, 36	tion.	
Limestone, as precipitant in leaching, use of...	44, 45	Roasting and leaching of sulphide ores, cost of	139
Lyon, D. A., work of	9	Roasting and leaching process, costs in	118
M.		description of	118
Malm process, attempted use of	123	Roasting and leaching tests, results of	127-129
Marsh mill (Idaho), zinc sulphide concentrate		S.	
from, analysis of	141	St. Francois mill (Mo.), lead-iron sulphide	
Mary Murphy mine (Colo.), zinc sulphide		middlings from, analysis of	160
concentrate from, analysis of	141	Salt, common, cost of	108, 110
roasting and leaching test of	143	consumption of in roasting and leach-	
May Day mine (Utah), oxidized ore from,		ing tests	127
analysis of	13	Salt Lake City station, investigations at	10, 12
brine leaching of	20-24	Schwarz, Alfred, on flotation of oxidized min-	
cyclic leaches of	53	erals	85
association of lead in	15	Scranton mine (Utah), oxidized ore from,	
screen analysis of	18	analysis of	13
sulphidizing and flotation, results of ..	89, 104	brine leaching tests of	24, 25, 30
May Kirby mine (Nev.) oxidized ores from,		lead in, association of	15
analysis of	13	sulphidizing tests of, results of	98
Metallizing of lead for flotation	105	Screen analyses of oxidized lead ores	18, 19

	Page.		Page.
Sells mine (Utah), oxidized ores from, analysis of.....	13	Sulphur vapor as sulphidizing agent.....	104
chloride volatilization of, results of.....	79	Sulphureted oil, as flotation agent.....	104
Shattuck mine (Ariz.), oxidized ores from, analysis of.....	13	Sulphuric acid, cost of.....	110
cyclic leaches of, results of.....	56	consumption of, in cyclic leaches.....	41, 42
sulphidizing tests of, results of.....	97	T.	
Silver, in calcines, extraction of by brine leaching, difficulties in.....	136	Tailing from cyclic leaches, analyses of.....	42
in low-grade ores, recovery of.....	16	Tailings, oxidized, tested.....	14, 15
in oxidized ores, extraction of by brine leaching.....	113	Terry, J. T., sulphidizing process of.....	85
by chloride volatilization.....	76, 79	Timber Butte mill (Mont.), zinc sulphide concentrate from, analyses of.....	141
by chloridizing roasting.....	66	Tintic district, low-grade lead ore in, amount of.....	15
by cyclic leaches.....	53, 54, 55, 56	Trapper mill (Mont.), oxidized tailing from, chloride volatilization of, results of.....	78
by roasting and leaching.....	62, 63	Trona, as precipitant in cyclic leaches.....	44
by sulphidizing and flotation.....	89, 90, 91, 97, 99, 100, 104	U.	
by volatilization and leaching.....	73	Udy, M. J., work of.....	36
volatilization of, reactions in.....	80	Utah, low-grade copper ores in.....	9
Silver-bearing lead carbonate ores, chloridizing roasts of.....	66, 67	low-grade lead ore in, amount of.....	15
roasting and leaching of.....	58-67	University of, cooperative work of.....	9-11
volatilization and leaching of.....	73, 74	V.	
Slimes, lead carbonate, flotation of.....	84	Volatilization, chloride, conditions of.....	80, 81
oxidized, tested.....	14, 15	Volatilization process, advantages of.....	117, 119
sulphide, galena in, recovery of.....	123	applicability of.....	169
Sodium carbonate, as precipitant in cyclic leaches.....	44, 49	chemicals for, cost of.....	115
Sodium chloride, effect of in roasting and leaching.....	64	possible application of.....	83
melting point of.....	80	tests of results of.....	70-72
See also Salt, common.		See also Chloridizing roast, roasting.	
Sodium sulphate, cost of.....	120	Volatilization and leaching process, advantages of.....	118
effect of on solubility of lead sulphate.....	31	costs in.....	119
Sodium sulphide, as precipitant in cyclic leaches.....	44, 49	factors in.....	75
as sulphidizing agent, application of.....	94, 101, 102	fume from, treatment of.....	83
cost of.....	94	W.	
melting point of.....	94	Wells, A. E., cited.....	9
sulphidizing action of.....	95	Wigton, G. H. work of.....	75
sulphidizing tests with.....	95-102	Wilbert mine (Idaho), oxidized ore from, analysis of.....	13
Sodium sulph-hydrate, as sulphidizing agent, use of.....	94	brine leaching tests of.....	25, 28, 29
South Hecla mine (Utah), oxidized ore from, analysis of.....	13	efficiency of, figures showing.....	28
Sulman, H. L., and Pickard, H. F. K., sulphidizing process of.....	85	lead in, association of.....	136
Sulphates in brine, effects of.....	31-33	roasting tests of.....	64, 65
Sulphide ores, complex, roasting and leaching of.....	125, 139-159	screen analysis of.....	19
treatment of, processes for.....	123-169	Williams, C. E., work of.....	9
Sulphidizing and flotation process, advantages of.....	120	Winninghoff, W. J., work of.....	31
concentrate from, value of.....	121	Y.	
costs of.....	120	Yellow Pine mine (Nev.), oxidized ore from, chloride volatilization of, results of.....	78
plant for, cost of.....	121	Z.	
Sulphidizing of carbonate ores, interfering elements in.....	101	Zinc, in lead-iron sulphide middlings, extraction of.....	163
of oxidized minerals.....	85-106	in lead sulphide ores, penalties on.....	139
Sulphidizing agents, merits of.....	106	in oxidized ores, extraction of, by chloride volatilization.....	77, 78, 79
Sulphidizing tests, conclusions on.....	106	in cyclic leaches.....	53, 54, 55, 56
See also Hydrogen sulphide, calcium polysulphide, sodium sulphide.		by roasting and leaching.....	63
Sulphur, boiling point of.....	104	by sulphidizing and flotation.....	90, 91
colloidal, as flotation agent, possible use of.....	105	in complex sulphide ores, recovery of, by roasting and leaching.....	164
cost of.....	104	in sulphide concentrates, extraction of, by roasting and leaching.....	147, 151, 155
effect of, in chloridizing ores.....	81, 82	by volatilization.....	150
		Zinc blende concentrates, analyses of.....	141
		Zinc ores, volatilization of, difficulty in.....	83
		sulphide concentrates, analysis of.....	141
		roasting and leaching tests of.....	142, 167
		Zinc sulphide, leady, roasting and leaching of.....	140
		cost of.....	140, 159

U.S. Mines, Bureau of,
Bulletin 157.

NAME OF BORROWER.

July 12/1948 G. Caranza 602 Crawford St.
p. 105218

